SWEENEY—

Extraction of Soybean Oil by Trichloroethylene

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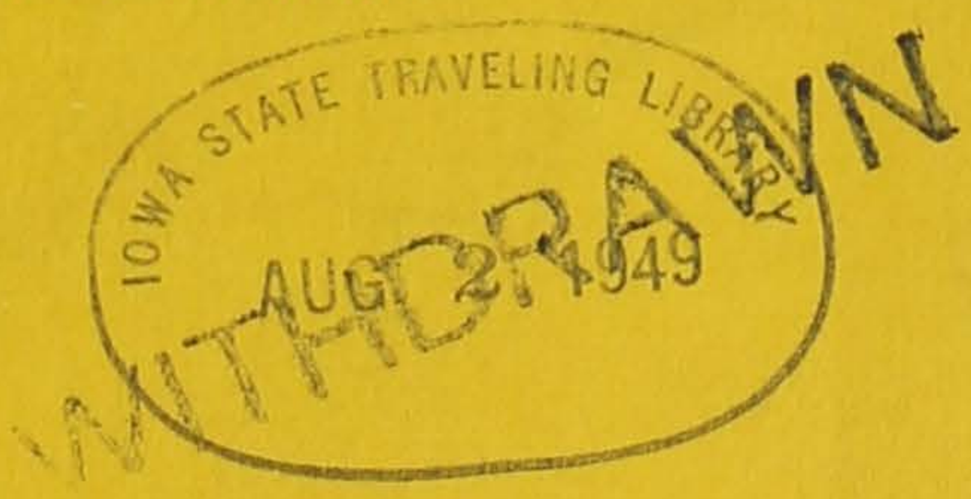
BULLETIN 165

EXTRACTION OF SOYBEAN OIL BY TRICHLOROETHYLENE

by

O. R. Sweeney, L. K. Arnold
and E. G. Hollowell



1949

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J. D. RYDER, *Assistant Director*

Bulletin 165
of the
IOWA ENGINEERING EXPERIMENT STATION

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by

O. R. SWEENEY
Research Professor of Chemical Engineering

L. K. ARNOLD
Research Professor of Chemical Engineering

E. G. HOLLOWELL
formerly
Research Associate of Chemical Engineering

THE IOWA STATE COLLEGE BULLETIN
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ACKNOWLEDGEMENT

It is impossible to give complete credit to all persons engaged in carrying out a research project of this type. The work on the development of the first pilot-plant unit was carried out in 1937-39 under a fellowship established by E. I. duPont de Nemours and Company. Subsequent work was financed by Iowa Engineering Experiment Station funds. A large number of students of Chemical Engineering, both undergraduate and graduate, have worked on problems connected with soybean processing and contributed to the success of this work.

ABSTRACT

An early survey of available non-flammable solvents indicated trichloroethylene to be a very promising agent for the extraction of soybean oil. Preliminary laboratory studies led to the design, construction, and operation of three successive pilot plants. The first utilized an extractor in which the flaked beans were carried by means of a screw conveyor down through an inclined tube in which trichloroethylene was flowing counter-current to the flaked beans. Stripping of the solvent from the oil was carried out in a falling-film evaporator. The extracted meal was dried in steam-jacketed pipes through which it was moved by modified screw-conveyors.

The second unit consisted of a modification of the first system in which the stripping was carried out in one end of the extraction tube. In the third plant, the flakes were carried through the extractor by a Redler conveyor and this proved to be the most satisfactory design. While a stripper was built originally into one end of the casing in this extractor, the most satisfactory stripping was obtained in a combination of a climbing-film evaporator and a packed-column stripper. A commercial plant with a capacity of about 15 tons per day was designed by the authors and put into successful operation in cooperation with a commercial feed mill.

A very sensitive method for detection of small amounts of trichloroethylene in soybean oil was also developed.

Patent applications have been filed covering certain features of the apparatus described, and equipment is being manufactured under a license given to the Crown Iron Works Co., Minneapolis, Minn.

EXTRACTION OF SOYBEAN OIL BY TRICHLOROETHYLENE

INTRODUCTION

Economic considerations seem to indicate that small-scale soybean-oil extraction plants located near the sources of soybean supply, in the smaller farm communities, should have certain advantages over large-scale extraction units concentrated in the cities. Such small plants would remove the oil from the beans for sale to industries as crude soybean oil, and would sell the bulky residue locally as soybean meal for feeding to livestock as a protein supplement. The advantages of the small local plant stem largely from low transportation costs since the beans originate on farms nearby and the bulky soybean meal may be sold to local livestock feeders. Only the concentrated, high-value oil need involve more than shipment by local truck.

To be successful such a plant must be efficient, economical, and safe in operation. Consideration of available information in regard to soybean-oil plants points to the conclusion that solvent-extraction plants offer a better opportunity for the development of an efficient small plant than pressure plants. Since soybeans have a relatively low oil content — about 20 per cent (see Tables 2 and 3) — and since the oil is the higher priced of the two end-products, it is desirable to obtain a high degree of oil recovery. Pressure plants leave about 5 per cent of the oil in the meal, solvent plants about 1 per cent. Thus the pressure plants remove only about 75 per cent, while the solvent plants remove about 95 per cent, of the original oil. At current prices* this represents

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* Calculations based on the following assumptions: Beans 18.2 per cent oil (20 per cent on dry basis) and 10 per cent moisture, meal 11 per cent moisture. Manufacturing loss considered the same for both processes and therefore neglected. Oil price 28 cents per pound and soybean meal $90 per ton (price from Chicago Journal of Commerce, June 1948). Weight of beans per bushel 60 pounds. Oil content of pressure-produced meal 5 per cent, solvent 1 per cent (on 11 per cent moisture basis). Difference in oil content 2.1 pounds per bushel, meal content 2.3 pounds per bushel. The above prices which were current when this manuscript was being prepared are considered high, but the difference for prices current at any time may be calculated by the following formula

Differential = price of oil in cents per lb. $\times$ 2.1 — price of meal per lb. $\times$ 2.3

Pressure-produced meal containing less than 5 per cent and solvent-extracted meal containing less than 1 per cent oil can be produced, but it is believed that the above figures are comparative for average conditions.

TABLE 1.
SOYBEANS: GRADE REQUIREMENTS*

Grade No.	Minimum test weight lb. per bu.	Maximum limits			
		Moisture Per cent	Splits Percent	Damaged kernels Per cent	Foreign material other than dockage Per cent
1	56	13	10	2	1
2	54	14	15	2	2
3	52	16	20	5	3
4	49	18	30	8	5

Sample grade includes beans not in above classification; musty, sour, heating, or hot soybeans; beans having a commercially objectional foreign odor; or otherwise of distinctly low quality.

Yellow and green soybeans of grade 1 may not contain more than 2 per cent, and grade 2 not more than 3 per cent black, brown or bi-colored beans.

* From Official Grain Standards of the United States for Soybeans, May 31, 1941. U. S. Dept. of Agriculture.

a difference of about 48 cents per bushel in the returns. Even at the relatively low price of 5 cents per pound for the oil and $20 per ton for meal there is a differential of 8.2 cents per bushel.

If processing costs are equal it appears that a solvent-extraction plant is likely to be a more profitable venture than a pressure plant. Solvent-extracted meal has a high protein content, and since soybean meal for animal feeding is sold primarily as a protein supplement, this high protein content should make the solvent-extracted meal desirable. In the solvent process the meal may be subjected to a rather wide range of temperatures to produce the desired type of product since extraction efficiency, unlike that in the pressure processes, is independent of this heat treatment. Thus for industrial uses, such as glue, plastics, or synthetic fibers, a light colored meal dried at a low temperature may be produced. For food purposes the meal should be given further heat treatment to produce maximum palatability and digestibility. The low oil content of the solvent-extracted meal makes it better adapted for use in industrial products than the pressure-produced meal.

Prior to the beginning of the present studies all of the solvent-extraction plants for soybean oil in this country were relatively large — about 100 tons per day or larger — and used commercial hexane* as a solvent. Hexane, while a good solvent, is flammable and its vapor forms explosive mixtures with air. Since

* A special cut of gasoline consisting of hexane, mixed with other hydrocarbons.

TABLE 2.
COMPOSITION OF SOYBEANS, PER CENT

Constituent	Average of 10 varieties planted at 5 locations[2]	Average of 43 varieties[3]	Average of many varieties[4]
Moisture	0	0	8.0
Protein[1]	42.87	39.83	40.0
Oil (Fat)	19.63	21.29	18.0
Crude Fiber	5.52	5.36	3.5
Ash	4.99	5.32	4.6
Total Sugar	7.98		
Sugars			7.0
Pentosans			4.4
Starch-like substances			5.6
Carbohydrates		27.41	
P_2O_5			1.7
K_2O			2.3
CaO			0.5
MgO			0.5

[1]—Protein = Nitrogen × 6.25.

[2]—Average of 10 varieties each grown at Ames, Iowa; Columbia, Missouri; Urbana, Illinois; Lafayette, Indiana; Columbus, Ohio, from: Carter, J. L. and Hopper, T. H. Influence of variety, environment, and fertility level on the chemical composition of soybean seeds. U. S. Dept. of Agriculture, Technical Bulletin 787, May, 1942.

[3]—Averaged from data given in: Mooers, C. A. The Soybean. Agricultural Experiment Station, Tennessee, Bulletin 82, 1908; Piper, S. V., and Morse, W. J. The Soybean. McGraw-Hill, 1923; Satow, S. Research in Oil and Proteids, Extraction from Soybeans. Sendai, 1921. Reprinted from Technical Report, Tokio Imp. University. Volume 2, No. 2.

[4]—Bailey, L. H. Soybeans, soybean flour, and soybean bread. Cereal Chem. 12, No. 5. September, 1935.

a small plant could not economically carry as overhead the cost of a staff of technical and safety men, it was considered important that the plant developed be operable without unusual safety precautions. This definitely ruled out the use of flammable liquids such as hexane, benzene, and carbon disulfide, and made it necessary to use a non-flammable solvent.

The problem was then to develop the design and operating technique for a solvent-extraction soybean oil plant which would have the following characteristics:

1. Suitable for operation in small sizes—10 to 20 tons per day in capacity.
2. Efficient.
3. Simple in construction and operation.
4. Using a non-explosive solvent.

In working out this problem, three complete extraction units together with necessary solvent-recovery apparatus were set up in sequence in the laboratory. Since the problems involved in the

TABLE 3.
AVERAGE COMPOSITION OF SOYBEAN VARIETIES*

Variety	Years	No. of samples averaged	Composition (dry basis)		
			Protein per cent	Oil per cent	Iodine No.
Habaro	1943-46[1]	39	43.3	18.8	133
Earlyana	1943-46	39	42.5	19.7	135
Wis. Manchu #3	1943-46	39	41.2	19.9	135
Earlyana	1940-46[2]	107	43.4	20.2	132
Richland	1940-46	107	41.0	20.3	128
Mukden	1940-46	107	43.6	19.5	130
Lincoln	1940-46	107	40.0	21.0	136
Dunfield	1940-46	107	40.0	20.5	130
Illini	1940-46	107	40.7	19.9	134
Lincoln	1942-46[3]	84	40.3	21.5	135
Dunfield	1942-46	84	39.1	21.4	130
Chief	1942-46	84	40.1	20.6	133
Illini	1942-46	84	40.6	20.4	134

[1]—From northern part of soybean belt; States of Iowa, Indiana, Ohio, Wisconsin, Minnesota, Michigan and South Dakota.
[2]—From central part: Illinois, Iowa, Indiana, Ohio, Michigan, Nebraska and Wisconsin.
[3]—From southern part: Illinois, Iowa, Indiana, Ohio, Missouri, Nebraska and Kansas.
* Data from U. S. Regional Soybean Laboratory, Urbana, Illinois.

preparation of the beans for extraction, in the analytical and other control work, and in the choice of solvent, were common to all units they are discussed in separate sections. Brief sections on soybean characteristics and on processing methods in general are included to give a better understanding of the problem. Design features and operation of the commercial unit are also presented.

SOYBEANS AND SOYBEAN OIL CHARACTERISTICS

Soybeans are annual legumes native to China where they were cultivated prior to written history, and where they have formed an important source of protein food for many centuries. While soybeans have been known in this country for many years they have only recently come into prominence as a major farm crop. Soybean production increased in the United States in the 27-year period from 1919 to 1946 from approximately 2.5 to 197 million bushels. While soybeans may be grown in practically all states, over 85 per cent of the nation's production is in Illinois, Iowa, Indiana, and Ohio. Production in these states in 1946 was as follows: Illinois, 75,036,000 bushels; Iowa, 34,960,000 bushels;

Indiana, 25,346,000 bushels; and Ohio, 16,254,000 bushels (50).

Analyses of Beans

Soybeans vary in composition with variety, soil, and weather conditions but are considered essentially as a source of protein and oil. The results of analyses of soybeans from various sources are shown in Table 2 and the compositions of representative varieties are given in Table 3. The usual varieties of soybeans will average about 40 per cent protein and 20 per cent oil (dry basis), although beans containing as high as 27 per cent oil have been reported (28).

Physical Characteristics

Soybean oil from mature beans is yellow or yellow-brown in color, bleaching readily to a pale yellow. While it has a characteristic "beany" taste, it can be refined to a bland oil suitable for food uses. It is classed as a semi-drying oil, its drying characteristics varying considerably as evidenced by reported iodine numbers ranging from 103 to 151. Most commercial soybean oil, however, approximates closely an iodine number of 130. The range of viscosity values for the oil is indicated in Fig. 1 and the specific heat as a function of temperature is given in Fig. 2. Other characteristics of the oil are given (12) as

	Pressure-produced oil		Solvent-extracted oil	
Average smoke point	183°C.	(361°F.)	210°C.	(410°F.)
Average flash point	300°C.	(572°F.)	316°C.	(601°F.)
Average fire point	351°C.	(664°F.)	355°C.	(671°F.)

Composition

Soybean oil contains the glycerides of both saturated and unsaturated fatty acids. Saturated-acid glycerides constitute approximately 12.5 to 15.0 per cent and unsaturated-acid glycerides 85 to 87.5 per cent of the total oil. The unsaturated acids are approximately as follows: oleic acid 21 to 34 per cent, linoleic acid 46 to 59 per cent, and linolenic acid 2 to 10 per cent. The saturated acids are mainly palmitic (about 6.7 per cent), stearic (about 4.3 per cent), and arachidic (about 1 per cent).

Also present in the oil are phosphatides, ordinarily in amounts of 1.5 to 2.5 per cent, in addition to small amounts of other constituents such as sterols and pigments. The phosphatides present in the oil are mainly cephalins and lecithins. Lecithin is usually considered a glyceride in which one of the fatty acid radicals is replaced by a phosphoric acid radical and a choline

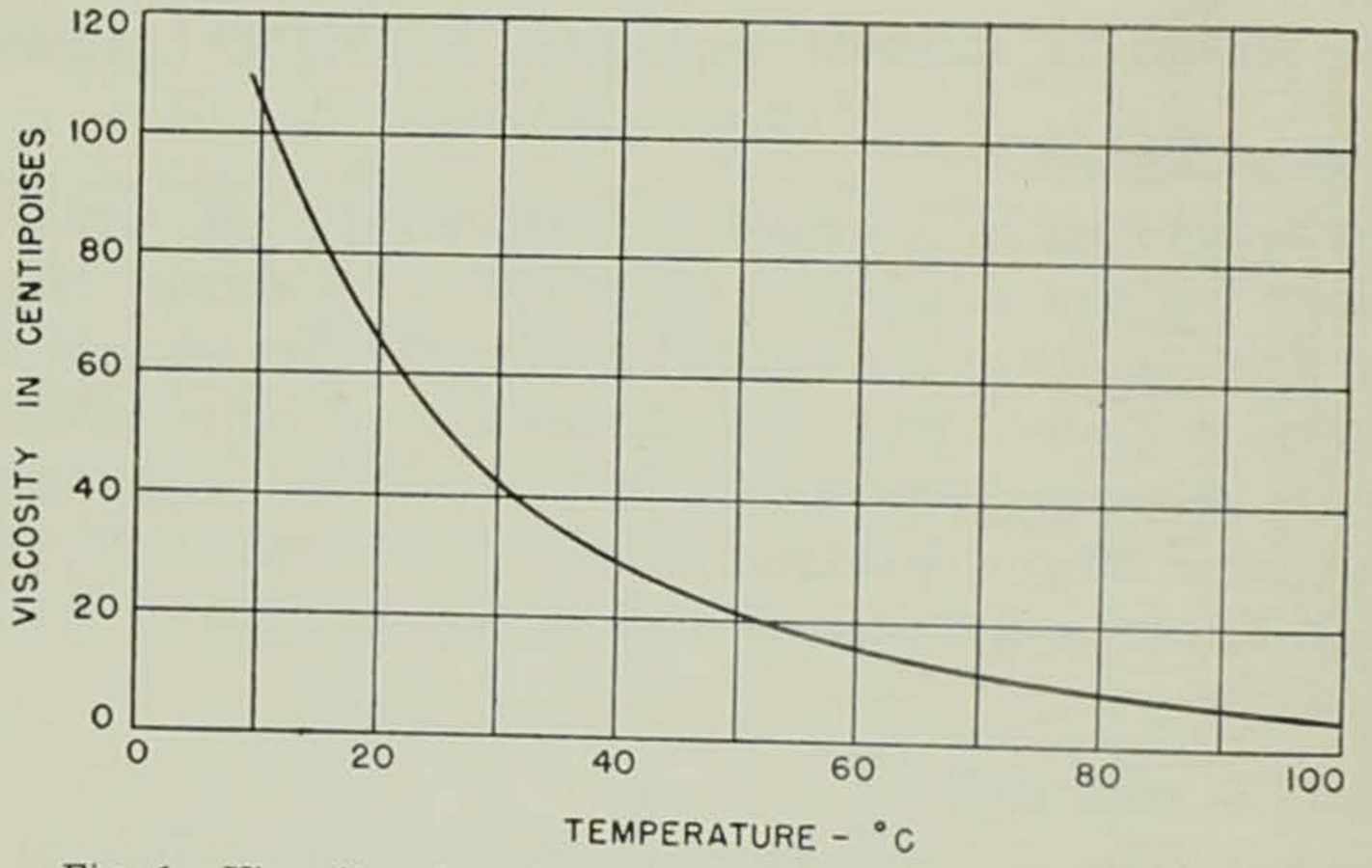

Fig. 1. Viscosity of soybean oil as a function of temperature.

radical. Since there are several fatty acids in soybean oil which may be present in various combinations, and since the phosphoric acid radical may occupy either the alpha or beta position, there are many lecithins possible. The phosphatides are readily separated from the oil by the addition of water, coming out rapidly with hot water. The mixture of phosphatides is commonly referred to as "lecithin" and unless purified ordinarily contains oil as well as minor impurities.

Uses of Soybean Oil

The largest use of soybean oil (about 75 per cent of the pre-war consumption) is in food products such as cooking fats, salad oil, and margarine. Oil used for food purposes is refined by treating with caustic soda, bleached with diatomaceous earth and carbon, and deodorized by steaming under vacuum. Most of it is also hardened by treatment with hydrogen. The non-food uses of soybean oil are in paints and enamels, in core oils for foundries, in printing inks, in linoleum, and in soap. In paints soybean oil is usually mixed with other drying oils although by the use of proper driers it may be utilized alone. Recent research indicates that soybean oil may be split into two fractions, one of high iodide number suitable for paint, and one of low iodine number which is better adapted for food uses than the original oil (43).

The lecithin obtained is an excellent emulsifying agent and has numerous commercial uses. It is used in margarine, vegetable shortenings, chocolate coatings, in pharmaceutical and cosmetic preparations, in leather tanning, in textile work, and in soaps.

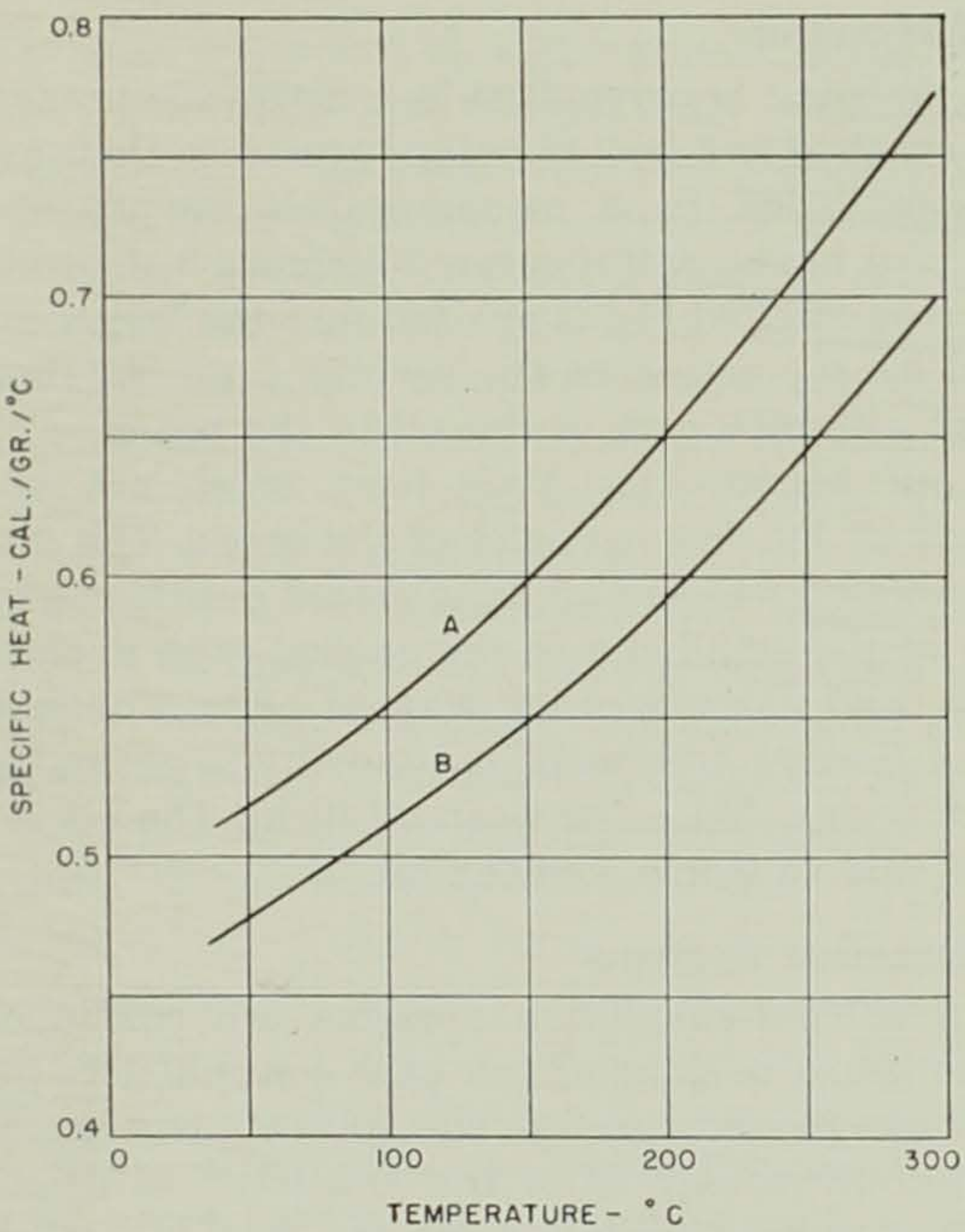

Fig. 2. Specific heat of soybean oil as a function of temperature. Curve A from reference (33), Curve B from reference (11).

Soybean Oil Meal

The residue remaining after removal of the oil is called soybean-oil meal. Meal produced by pressure methods will average about 5 per cent oil and 44 per cent protein on a dry basis. Solvent-extracted meal contains about 1 per cent oil and 46 per cent protein. The meal is usually sold containing about 11 per cent moisture. The largest use for soybean-oil meal is as a protein supplement in the feeding of farm animals. In the form of grits and flour it is incorporated into many human foods such as bread, muffins, macaroni, sausages, pancake flour, and various pastry items.

METHODS OF OIL EXTRACTION

Oil may be removed from oil-bearing seeds, such as soybeans, by pressing or by dissolving with a suitable solvent. By either method the oil removal may be carried out as a batch operation or as a continuous operation.

Pressure Extraction

Batch pressing is carried on in a hydraulic press, the beans first being cracked and heated before pressing. Continuous pressing is accomplished in a mechanical screw-press, consisting essentially of a heavy spiral screw revolving in a conical cage of steel bars. Hot cracked beans are fed into the larger end and are compressed by the action of the revolving screw. Under the resulting high pressure part of the oil in the beans is pressed out and flows out between the steel bars which act as a screen, keeping back all but fine particles of the beans. The solid residue, containing about 5 per cent oil, is squeezed out through a restricted opening at the small end of the conical cage in the form of a hard, dense, and flat but much warped cake. The cake may be sold without further treatment but usually is ground to produce the product designated as soybean-oil meal. The oil is filtered in a press and sold as crude soybean oil.

Solvent Extraction Systems

Modern solvent-extraction systems are continuous in operation. The beans in flaked form pass continuously into the extractor and are passed through the solvent. The extracted meal is removed continuously from the extractor to driers, usually steam-jacketed tubes through which the meal is moved by screw or paddle conveyors, where the solvent is driven off as vapor and condensed in a surface condenser for re-use. The solvent-oil mixture or **"miscella"*** is drawn off continuously from the extractor to a still or stripper where the solvent is volatilized and passed to a condenser. The various extractors which are in use may be considered in four groups as (1) basket type, (2) vertical tower type, (3) U or V screw conveyor types, and (4) horizontal types.

The basket, or Bollman system, one of the oldest of the continuous systems, was developed in Germany where it was used by the Hansa Company (20) (44). In this extractor a series of wire-mesh baskets are attached to an endless chain in a manner similar to a vertical bucket elevator. The baskets are suspended so that they remain open side up until they have made almost a complete revolution of the machine and are ready to dump. The flaked beans are fed into the baskets as they pass the upper position and the extracted meal is dumped out after the

* The word "miscella" is commonly used in industry to indicate a mixture of oil and solvent.

baskets have gone down and up again almost to the filling point. Fresh solvent is sprayed onto the partially exhausted flakes as they come into a position just below the dumping point. This solvent passes to the bottom of the extractor through the rising series of baskets resulting in counter-current extraction. The solvent, now containing considerable oil, is pumped from the bottom and sprayed onto the fresh flakes entering the extractor. This miscella passes down through the downward-moving series of baskets and, since it is moving in the direction of the basket, does not extract counter-currently.

In the vertical-tower extractor flaked beans are dropped into the top of a vertical tower or into an extraction column and allowed to pass downward through the solvent to the bottom. The towers are ordinarily equipped with a series of shelves having staggered openings through which the meal drops. The meal is scraped around to the opening on each shelf by a revolving scraper-arm. A variation of this form uses revolving shelves and stationary scrapers. The solvent enters at the bottom of the tower and the miscella is drawn off at the top. In the original Bonotto system (5) the meal was taken out at the bottom of the tower by a screw device which squeezed the miscella from the meal much as oil is squeezed from the beans in a mechanical screw-press. A common method at present is to take the meal out with a conveyor of the Redler or similar type, carrying it in a tight casing to a point high enough above the tower to secure good drainage and then dropping it into a drier. Another method is to remove the flakes from the bottom of the tower by a vertical screw conveyor operating in a tube extending down the center of the tower and discharging out through the side of the tower (3).

In the Boehm system (20) two series of screen disks are attached to revolving vertical shafts in such a manner that the disks of one series alternate in vertical positions with those of the other. These disks are partially enclosed with solid cylindrical sides and are so placed that the top disk of one series discharges into the top disk of the other series, which in turn discharges into the second one of the first series. The flaked beans are scraped off each screen by a stationary arm. Fresh solvent is pumped onto the beans on the bottom screen. After passing through the bottom screen, it is pumped up and sprayed

on the screen above. This procedure is repeated to the top thus producing counter-current extraction.

Instead of dropping the flakes through the solvent they may be carried downward by a screw conveyor. One of the earliest models of this type of extractor was the Hildebrandt system (38), originally built in Switzerland and later introduced into this country. In this system two large vertical tubes are connected across the bottom to form a U, with the flakes carried by screw conveyors. The flaked beans enter at the top of one leg of the U and the extracted meal is taken out of the top of the other leg. The solvent enters near the top of the leg from which the meal is removed, and the miscella flows out just under the point where the flakes enter, thus giving counter-current extraction. A variation of this is an extractor using two tubes with screws —one vertical and one at an angle (24). Flaked beans are fed into the top of the vertical leg and pass downward to the bottom end of the sloping leg. The extracted meal comes out at the upper end of the sloping leg, with counter-current solvent flow. Another extractor is designed as a long narrow rectangular chamber, the bottom of which consists of a series of semi-circular sections each containing a perforated paddle. The flaked beans are fed in at one end and are moved towards the opposite end from section to section by the paddles, and after extraction are removed by an inclined chain conveyor. The solvent enters at the point of removal of the flakes and the miscella flows out at the opposite end (25). All of the above systems use commercial "hexane" as a solvent.

In a recent system developed after the work in this bulletin was begun, and using trichloroethylene as a solvent, the flaked beans are carried through the extractor horizontally by a screw from which they are elevated at one end to screw-type tubular driers. The flow is counter-current, the miscella being taken off through a screen above the screw.

Pressure Versus Solvent Processing

At the beginning of the work reported in this bulletin the advantages and disadvantages of the two methods could have been summarized as follows:

Pressure Methods

Advantages —

1. Equipment well designed mechanically.

2. Equipment available in units of 15 tons per day and larger.
3. Operation standardized and not highly complicated.

Disadvantages—

1. High residual oil content in cake — over 5 per cent.
2. High repair and depreciation costs because of high pressures employed.
3. High power requirements.

Solvent-Extraction Methods

Advantages —

1. Low residual oil content in meal — 1 per cent or less.
2. High protein content in meal.
3. Meal may be heated to any desired temperature without change in the extraction efficiency.
4. Low power requirements.

Disadvantages —

1. Available only in larger sizes — 100 tons per day or over.
2. Equipment not well developed — still somewhat experimental.
3. Operation rather complicated.
4. Hazard from use of flammable solvents such as hexane.

Since these studies were started solvent-extraction systems have been improved and small units using non-flammable solvents are being offered.

THE CHOICE OF A SOLVENT

There are many solvents for soybean oil, some of which have been used commercially with varying success. In addition to being a good solvent for oil, certain other qualities are desirable in a solvent if it is to be suitable for industrial use. It must not damage the oil and meal, and not leave any residues in them. It is desirable that it be: non-explosive, non-corrosive to the common metals, non-poisonous, readily available, and low in cost.

General Characteristics

Prior to the beginning of this investigation commercial

"hexane" was the solvent used in practically all soybean oil-extraction plants in the United States. This solvent is a special cut of gasoline having a narrow boiling range and a volatility such that it is readily removed from both the oil and meal. Ordinary motor fuels are not satisfactory for this purpose since the heavy components which they contain are difficult to separate from the oil and meal as was demonstrated in early work in this laboratory when ordinary motor gasoline was used. Distillation of a mixture of gasoline and oil began at 110°C. (230°F.) and continued up to about 180°C. (356°F.) at which point the residue in the still was less than 50 per cent oil, the remainder being heavy ends which could not be distilled out and which began to crack at about 180°C. (356°F.). At this temperature the soybean oil had commenced to decompose making further distillation impractical. Some of the earlier solvents which the hexane has displaced, such as carbon disulfide, ethyl ether, and acetone, were found to be less desirable because of greater fire hazard and greater cost.

Since the present extraction studies were concerned only with non-flammable and non-explosive solvents, hexane was not considered for possible use in the extraction process being developed. It is of interest, however, to consider it in comparison with the other solvents studied since it is the principal solvent being used in other extraction processes.

The non-flammable solvents for vegetable oils, available in commercial quantities, are found largely among the halogenated hydrocarbons, particularly the chlorinated compounds. When the hydrogen of a hydro-carbon is replaced by a halogen such as chlorine, the flammability of the compound is reduced. If sufficient hydrogen atoms can be replaced by chlorine, the new compound may become non-flammable. This is well illustrated in the reduction in flammability of chlorine compounds of methane with increased chlorine content. Carbon tetrachloride, the result of the substitution of chlorine for all of the hydrogen atoms, is classed as of zero flammability and is used as a fire extinguishing liquid. In addition to the chlorinated derivatives of methane those of ethylene and ethane are of interest as possible soybean oil solvents. The physical properties of some of the more promising of these chlorinated hydrocarbons, together with those of hexane, are given in Table 4.

TABLE 4

PHYSICAL PROPERTIES OF VARIOUS SOLVENTS

	Methylene Chloride	Chloroform	Carbon tetrachloride	Trichloro-ethylene	Perchloro-ethylene	1,1,2-Tri-chloroethane	Ethylene dichloride	Hexane normal
Chemical formula	CH_2Cl_2	$CHCl_3$	CCl_4	CHCl CCl_2	CCl_2 CCl_2	CH_2Cl $CHCl_2$	CH_2Cl CH_2Cl	CH_3 $(CH_2)4$ CH_3
Molecular weight	84.9	119.4	153.8	131.4	165.8	133.4	99.0	86.1
Boiling point (at 1 atm) °C.	39.8	61.2	76.5	86.9	121.2	113.7	83.5	*60-71°C ‡69°
Steam distillation point (at 1 atm) °C	38.3	56.0	66.8	73.2	87.7	85.4	71.4	
Latent heat of vaporization (at b.p.) cal./gr.	78.7	59.3	46.8	57.2	50.0	68.7	77.3	†79.3
Specific heat, liquid cal./g./°C - 20°C.	0.280	0.234	0.205	0.225	0.205	0.266	0.308	†0.600 20-100°C.
Specific gravity of liquid	1.326	1.489	1.594	1.464	1.623	1.438	1.253	‡0.6603
Specific gravity of vapor 1 atm. and b.p. air	2.93	4.13	5.32	4.54	5.83	4.61	3.42	2.97
Viscosity, liquid 20°C. centipoises	0.425	0.57	0.969	0.58	0.88	1.2	0.84	§0.71
Solubility in water 25°C. g./100g.	1.32	0.79	0.08	0.11	0.015	0.44	0.84	(15) ‡0.0138
Solubility water in solvent 25°C. g./100g.	0.198	0.097	0.013	0.032	0.0105	0.16 (20°C)	0.025 (-9°C)	

Properties of chlorinated compounds from: McGovern, E. W. Chlorohydrocarbon Solvents. Ind. Engr. Chem. (35:1230(Dec. 43).
*—Skelly-Solve B data from manufacturer's literature.
†—Perry, J. H. Chemical Engineers' Handbook 1st Ed. McGraw Hill, 1934.
‡Hodgman, C. D. Handbook of Physics and Chemistry 25th Ed. Chemical Rubber Pub. Co. 1941-42.
§Johnstone, H. F., Spoor, I. H., and Goss, W. H. Properties of Soybean Oil-Solvent Mixtures. Ind. Engr. Chem. 32:832-835, June, 1940.

The primary requirement of any solvent for a specific oil is that it will readily dissolve the oil. Any ability of the solvent to dissolve non-fat materials is commonly considered detrimental. This is not necessarily true, but is a matter which must be considered for each case in question. The action of the solvent should not be such as to injure the residue. Solvent-processed meal may be readily heated either during the drying operation for removal of solvent, or in a subsequent operation to give the desired heat treatment for proper nutrition. It is not necessary that solvent meal be of a decided brown color, as is most pressure-produced meal, to be properly heat treated for good nutrition. The dark color of the pressure-produced meal is caused by the high temperatures necessary for maximum oil removal. Part of the color is due to fine particles of black partially-carbonized meal which have been overheated because of the high temperatures developed by friction of the screw on the meal.

Chlorinated Hydrocarbons as Solvents

The chlorinated hydrocarbons in general are good solvents of fats and oils. Several chlorinated hydrocarbons and commercial hexane were compared in their solvent action on flaked soybeans in laboratory Soxhlet extractors. All extractions were for 2-hour periods and were made on one batch of flaked soybeans which were stored in a tight container to prevent moisture changes. From three to four extractions were made in duplicate with each solvent, using two to three solvents per day. The extracted-oil percentage values were corrected on a comparative basis so as to eliminate as far as possible any errors due to variations which, according to Krober and Collins (27), may occur because of humidity variations. The average of the corrected values were as follows: hexane, 19.0 per cent; methylene chloride, 19.1 per cent; ethylene dichloride, 18.9 per cent; carbon tetrachloride, 19.2 per cent; chloroform, 19.7 per cent; and trichloroethylene, 19.8 per cent. In order to compare the effectiveness of these solvents on materials of lower oil content, pressure-processed meal was extracted by a procedure similar to that used for the beans. The following data on percentage extraction were secured: commercial pentane, 3.9 per cent; commercial hexane, 4.2 per cent; methylene chloride, 5.8 per cent; ethylene dichloride, 4.9 per cent; and trichloroethylene, 5.2 per cent. These data are

also of interest in connection with the possible use of these solvents in the determination of oil in meal in the analytical laboratory.

The quality of an oil produced by a solvent is important. Sievers and McIntyre (46) extracted oil from soybeans, peanuts, corn, and cottonseed with various solvents. The percentages of free fatty acids in the crude oil extracted by the various solvents from soybeans were: ether, 0.73; benzol, 0.86; carbon tetrachloride, 0.64; trichloroethylene, 0.64; light gasoline, 0.51; and heavy gasoline, 0.49. The refining losses in per cent, as determined by the standard laboratory method for the soybean oil extracted with the various solvents were: ether, 4.94; benzol, 8.45; carbon tetrachloride, 7.6; trichloroethylene, 8.3; light gasoline, 8.4; and heavy gasoline, 8.8. The average refining losses for all four oils were: trichloroethylene, 6.46; carbon tetrachloride, 7.18; light gasoline, 7.6; benzol, 8.26; heavy gasoline, 8.7; and ether, 10.21 per cent. Trichloroethylene-extracted soybean and peanut oil were the darkest oils but when refined gave the lightest oils of the group. These data indicate that the two chlorinated hydrocarbons, trichloroethylene and carbon tetrachloride, should produce soybean oil at least equal in quality to that produced by benzol or gasoline.

The quality of the meal produced by a given solvent is equally important. Solvent-extracted meal is sometimes said to be less nutritious and less tasty as an animal food than the meal produced by pressure systems. This criticism was doubtless justified in the case of some of the earlier solvent-extracted meal. In some cases, due to improper methods of production or the use of a solvent containing high boiling fractions, some solvent was left in the meal, but improvements in manufacturing methods have eliminated this undesirable feature. It is also possible that some of the meal may not have been heated to a temperature sufficiently high to give maximum food value. Research (15) (54) has shown that expeller meals processed at 140° to 150°C. (284° to 302°F.) for 2½ minutes, hydraulic press meals cooked at 105° to 121°C. (221° to 250°F.), for 90 minutes, and solvent-extracted meals heated at 98°C. (208°F.), for 15 minutes have approximately twice the nutritive value of raw soybeans or meals produced at low temperatures.

The ease of removal of the solvent from the oil and meal is

important. In general, definite chemical compounds such as the chlorinated hydrocarbons, having a definite boiling point, should be less difficult to remove than mixtures such as commercial hexane, having a boiling range. A low boiling point is a definite advantage as it allows removal of the solvent at a low temperature with less danger of injury to the oil or meal. The chlorinated compounds listed in Table 4 are those having boiling points low enough to be given consideration. The lower boiling solvents offer advantages of better heat transfer in strippers and driers for a given steam temperature.

Chlorinated compounds may be unstable and under certain conditions may break down, forming acidic products which may be corrosive to construction materials such as steel. Trichloroethylene, perchloroethylene, and methylene chloride are the most stable over a considerable range of conditions such as temperature, moisture, and light, (36). Methylene chloride may be used in ordinary equipment in the presence of air, light, and dissolved water up to 60°C. (140°F.). Undissolved water at 60°C. may cause hydrolysis and consequent acidity. Properly stabilized trichloroethylene may be used up to 120°C. (248°F.) in the presence of air, light, and moisture in common construction materials. Perchloroethylene is stable to 140°C. (284°F.).

In the presence of air chloroform develops acidity upon continuous use. In the presence of water carbon tetrachloride hydrolyzes enough to corrode common metals. Ethylene dichloride hydrolyzes in contact with water at 80°C. (176°F.). Trichloroethylene hydrolyzes at its boiling point with water. Since in a solvent-extraction process for soybeans, water from the beans and from steam used in stripping the solvent from the oil will be in contact with the solvent, it would seem that the most suitable of the chlorinated solvents would be trichloroethylene, perchloroethylene, and methylene chloride.

Various stabilizers have been proposed for trichloroethylene to prevent the development of acidity. These include alkaloids, volatile amines, pyridines, and essential oils (13). Phenolic substances (9) and substituted phenols (13) have also been proposed. Aniline and triethylamine have been used commercially. The latter because of its boiling point (89.5°C., 194°F.) and less poisonous properties is the more suitable for soybean processing.

In the recovery operation the solvent vapors, together with

water vapor, are condensed and are reused after separation from the water. It is desirable that the solvent have a low solubility in water to facilitate separation with the minimum loss of solvent. Of the three solvents noted above trichloroethylene has the lowest solubility in water although it does not differ greatly from perchloroethylene. Perchloroethylene has the lowest heat of vaporization, an advantage more than offset by its boiling point. Methylene chloride with its low boiling point has the highest heat of vaporization. Trichloroethylene is the cheapest of the three solvents and is produced in large quantities for metal degreasing work.

Trichloroethylene vapor does not form flammable mixtures with air at ordinary pressures and temperatures. At high temperatures the vapors become flammable in air igniting at 463°C. (865°F.) (23). The limits of flammability for mixtures of pure oxygen and trichloroethylene vapors are 10.3 per cent trichloroethylene and 64.5 per cent, for the lower and upper limits.

Consideration must be given to the health hazards introduced by use of the solvents. Any liquid suitable for solvent extraction will evaporate sufficiently at room temperature to produce considerable vapor in the air. These solvent vapors, as is true of all vapors and gases other than air when inhaled in any appreciable quantities, produce abnormal physiological effects. While in any well-operated extraction system the amount of vapor normally escaping is very small, abnormal operating conditions may introduce vapors into the air in considerable amount. For this reason it is well to consider possible hazards. According to McGovern (36) the acute narcotic effects of high concentrations of carbon tetrachloride, ethylene dichloride, dichloroethylene, trichloroethylene, and perchloroethylene are of almost the same order of magnitude. Since carbon tetrachloride and ethylene dichloride have higher vapor pressures than trichloroethylene and perchloroethylene they represent somewhat greater hazards. It is also pointed out that the latter two do not produce delayed or cumulative physiological effects, such as liver damage, to the extent produced by the former. According to Lehman and Flury (30) the following are the highest safe concentrations for various solvents in the air of work rooms, in parts per million: benzine, (petroleum ether, mainly hexane) 200; benzol, 30; methylene chloride, 290; chloroform, 40; carbon tetrachloride, 30;

ethylene dichloride, 500; trichloroethylene, 190; perchloroethylene, 190. This indicates that ethylene dichloride has the lowest health hazard of the group with methylene chloride second, so that of the non-flammable solvents methylene chloride apparently presents the smallest health hazard. The low boiling point and high vapor pressure of the methylene chloride would probably offset this apparent advantage under practical conditions. Thus, there is little choice between the otherwise suitable non-flammable solvents. All present approximately the same health hazard as the flammable solvent benzine and much less hazard than the flammable solvent benzol.

After giving careful consideration to the properties, prices, and availability of the various non-flammable solvents, trichloroethylene was chosen as the most suitable for studies on soybean-oil extraction.

The use of mixtures of flammable and non-flammable solvents, such as hexane and trichloroethylene, was also considered as a possible means of securing a non-flammable solvent at a lower cost than with pure chlorinated compounds. Actually the cost advantage is not large because of the large amount of the non-flammable solvent needed in the mixture. For example, to secure a non-explosive vapor from a hexane-trichloroethylene mixture, it is necessary to have four parts of the latter by volume to one of the former (46).

Tests on Toxicity of Trichloroethylene-Extracted Meal

Several references exist in the literature to cases of poisoning of cattle by soybean meal made from beans extracted by trichloroethylene. Stockman (47) reports the death of several cows two years old or older after eating a ration containing trichloroethylene-extracted soybean meal for 29 or more days. The symptoms included external and internal hemorrhages, particularly in the small intestines and mucus membrane of the digestive tract and the lymph glands, black feces with a fetid odor, and a decrease in red blood corpuscles. Ten per cent of the cattle were visibly ill and of these 84 per cent died. Sheep and pigs were not affected by eating the meal. To determine whether or not the poisoning might have been caused by trichloroethylene remaining in the meal, cows were fed trichloroethylene added to their normal ration in doses ranging from one to three ounces a day for

long periods. The trichloroethylene failed to produce any poisonous symptoms. Similar trouble with trichloroethylene-extracted meal in Germany has been reported by Bleger and Meyer (4) Veillard (52) and Vartiainen, Itmari and Armas (51). The latter authors attribute the trouble to the presence of urease not destroyed in processing, and report the effect of urease on mice.

Studies on possible poisonous effects of trichloroethylene-extracted meal when fed to various animals were initiated in 1936 at Cornell University. The meal was first fed to rats, guinea pigs, and rabbits. The guinea pigs appeared sensitive to the meal showing evidence of hemorrhage at times, but the rabbits were unaffected. Eight weaned lambs were fed a mixture of 80 per cent grains and 20 per cent soybean meal extracted with trichloroethylene and processed in the range of 90°C. (194°F.). After being fed this ration for 5 months the lambs were slaughtered. The carcasses were government inspected and graded "A". Careful examinations indicated nothing abnormal. Ten pure bred Guernsey cows were then fed with concentrates containing 20 per cent trichloroethylene extracted meal processed at 90°C. for 4 months at the end of which time one cow died. An autopsy showed the same symptoms described by Stockman (47).

Ninety-six guinea pigs were next divided into groups and experiments conducted over a period of six weeks while feeding commercial pressure-processed soybean meal, trichloroethylene-extracted meal processed at 150°C. (302°F.) for 45 minutes, and methylene chloride-extracted meal processed at 60°C. (140°F.). The results secured with the six groups were as follows:

Group I. Three of five animals who received the trichloroethylene-extracted meal *processed at 150°C. (302°F.)* as 40 per cent of their total feed showed no abnormalities when killed at the end of four weeks. This was also true of the remaining two animals killed at the end six weeks.

Group II. Twelve of 24 animals fed trichloroethylene-extracted meal *processed at 150°C.* as 30 per cent of their total feed killed at the end of four weeks showed no abnormalities. The remaining twelve killed after six weeks likewise showed no abnormalities.

Group III. Twenty-four animals fed pressure-processed meal as in Group II showed no abnormalities.

Group IV. Twenty-four animals were fed the methylene

chloride-extracted meal *processed at 60°C. (140°F.)*. Thirteen died from the thirteenth to the thirty-seventh day. Four animals were killed at the end of four weeks and seven at the end of six weeks. Three of these animals showed hemorrhage in the lungs, gastro-intestinal tract, and muscles of the neck.

Group V. Of seven guinea pigs on normal diet four sacrificed at the end of four weeks and three after six weeks showed no pathology.

Group VI. Twelve guinea pigs were fed trichloroethylene extracted meal processed at 120°C (248°F.) as 30 per cent of feed. Six animals killed at the end of four weeks and six after six weeks showed no abnormalties.

Thirty-six additional animals were next fed, 18 with some of the same meal which had previously been used to feed the cows with apparent bad results, and 18 with trichloroethylene-extracted meal *processed at 60°C. (140°F.)* as 30 per cent of their diet. None of the first 18 killed at the end of four and six weeks showed pathology. The second group lost weight and became listless, four dying during the third week. Five animals killed at the end of four weeks and nine at the end of six weeks all showed hemorrhage in the lungs and muscles of hind quarters and pinpoint hemorrhage in the intestinal tract. As a result of this work with 132 guinea pigs, it was concluded that when trichloroethylene-extracted soybean meal was *processed at 120°C. (248°F.)* or above the toxic principle was destroyed.

As a further check on these conclusions trichloroethylene-extracted meal was fed to two herds of cows on two separate dairy farms. One herd contained 12 purebred Guernsey cows, the other 24 mixed cows divided into 12 experimental and 12 control cows. The ration used, Table 5, contained the trichloroethylene-extracted soybean meal *processed at 115 to 120°C. (248°F.)* for 30 minutes. This meal was produced at Iowa State College in the equipment described later in this bulletin.

Feeding experiments were run for a one-year period with the Guernsey herd, with the grain mixture being fed at the rate of 1 pound for each three pounds of milk produced, as a supplement to pasture. Dry cows received four pounds per day. In the fall alfalfa and clover hay and soybean silage preserved with phosphoric acid were fed in place of the pasture feed. The consumption of soybean meal per cow ranged from about 550 to 900

TABLE 5
CONCENTRATE MIXTURE USED IN EXPERIMENTAL FEEDING

100 pounds linseed oil meal	ANALYSIS:	
300 pounds wheat bran	Moisture	10.56
300 pounds hominy or corn meal	Protein	22.60
260 pounds ground oats	Fat	4.76
200 pounds corn distillers grains	Crude fiber	8.11
400 pounds soybean meal*	Ash	5.07
300 pounds coconut oil meal	Total digestible nutrients 75.2 per cent or 1503 pounds per ton.	
100 pounds molasses		
20 pounds steamed bone meal		
20 pounds salt		

* Trichloroethylene extracted meal produced at Iowa State College and processed at 115 to 120°C (239° to 248°F.) for 30 minutes.

pounds for the year. The average daily intake was from 1.4 to 2.7 pounds per day for a year as compared with 2.5 to 3 pounds per day for 26 to 70 days reported by Stockman (47) for cases of severe symptoms and death. All but one of the cows calved during the experiment. In general the health of the cows, as determined by monthly examination, was excellent. Periodic checks of temperature revealed no elevations. Reproduction, lactation, and milk and fat production were normal.

In the mixed herd one cow gave birth to twins, each having large goiters. This was traced to the fact that the cows were getting practically no iodine and had no evident relation to the experimental ration. Reproduction, lactation, and milk production were normal.

During the foregoing experiments both herds were under the supervision of specialists in veterinary medicine, animal nutrition, and animal husbandry. From these experiments *it is concluded that trichloroethylene-extracted meal processed at 115° to 120 C. (239 to 248°F.) for 30 minutes produces no bad effects when fed to dairy cattle* and is very satisfactory as a protein supplement. This is further shown to be true by the fact that about 17 tons of trichloroethylene-extracted meal produced experimentally in the present studies were fed to dairy cattle by the Dairy Industry Department of Iowa State College with entirely satisfactory results. Several tons were also fed by a local farmer to beef cattle.

The presence of a small amount of trichloroethylene in the meal apparently is not harmful. As has been mentioned Stockman (47) reports that doses of 1 to 3 ounces of trichloroethylene per animal added to the feed for a long period of time failed to cause

illness. Meal produced in the current studies, containing enough trichloroethylene to be plainly evident by odor, was fed to cattle with no apparent bad effects. Trichloroethylene evaporates rapidly from meal even when stored in bags, and will not remain present in meal stored for reasonable periods.

Soybean meal experimentally extracted under conditions similar to those used with trichloroethylene, but with methylene chloride and with ethylene dichloride as solvents, was fed in amounts of several hundred pounds to dairy cattle at Iowa State College without indicating any apparent bad effects from either meal.

Human Health Hazards Introduced by Trichloroethylene

As has been noted, trichloroethylene, in common with other solvents, presents certain health hazards. In the normal operation of a properly designed and operated solvent-extraction system, these hazards should disappear. As a guide to proper handling of the solvent, particularly under experimental or emergency conditions, it is well to be acquainted with the potential hazards.

In general, according to Henderson and Haggard (16), when inhaled the chlorinated hydrocarbons produce two effects: anesthesia and toxic action. The toxic action may affect such organs as the kidneys, the liver, or the nervous system. Trichloroethylene primarily affects the nervous system. In industrial work, anesthesia would occur only as the result of an accident in which the patient would be subject to a large concentration of the solvent vapor. Deep anesthesia might result in death from respiratory failure and death can also occur from fibrillation of the heart or possible damage to the nervous system. While several deaths from trichloroethylene have been reported in the literature (48), these apparently resulted from either gross or prolonged exposure, or in part from previous illness or from effects of other solvents mixed with the trichloroethylene. It is estimated (16) that 37,000 parts per million of trichloroethylene is necessary in the air to kill animals in a short time and 13,500 parts to cause deep narcosis in 30 minutes. Symptoms of acute trichloroethylene poisoning (18) are local irritation, tears, sleeplessness, headache, fatigue, pains in chest, shortness of breath, and ailments of the digestive organs.

Liquid trichloroethylene has an irritating effect on the skin,

causing burning and sometimes blisters (10). The amount of irritation varies with different individuals and upon different parts of the body of the same individual. The trichloroethylene removes the protective fat film from the skin thus making it more susceptible to secondary skin infections. It may also be absorbed through the skin to give effects similar to those caused by inhalation.

When trichloroethylene comes into contact with a hot metal surface or with fire it decomposes to form phosgene which is highly poisonous. Tests (2) show that when air containing 1000 parts per million of trichloroethylene was drawn through a lighted cigarette the inhaled mixture contained 3 parts per million of phosgene. This is sufficient phosgene to cause immediate irritation of the throat and is 3 times the amount allowable for prolonged exposure (16).

Protection against trichloroethylene consists largely in proper handling, and in keeping the extraction system tight to prevent leaks into the outside air. Any appreciable leakage can be detected by the odor and smaller amounts may be detected by a halide lamp in which a gas flame heats a copper disc. The flame burns green if a chlorinated hydrocarbon is present. Quantitative determinations can be made photoelectrically by a "Tri-Per" analyzer (36) which measures variations in light absorption by air containing solvent vapors.

If leakage occurs from equipment the health hazard may usually be minimized by good ventilation until the abnormal condition is corrected. In ventilating, the fact that trichloroethylene vapors are heavier than air, and therefore go downward in a room, must be considered.

Protection is given against a considerable concentration of vapor by the so-called paint respirators which contain activated carbon as an absorbent or by an industrial gas mask with a proper canister.

FLAKING STUDIES

Since in solvent extraction the time required for penetration of the solvent into the material and for diffusion out of the solvent-oil mixture is a function of the distance travelled, it is desirable that this distance be kept to a practical minimum. Reduction in thickness or particle size of the extracted material

also produces a larger surface area for contact with the solvent. For example, if soybeans are ground to a fine flour, they obviously present a large surface to the solvent, together with a short distance to be penetrated. This results in a short extraction time as has been shown experimentally by Yushkevish, Brilling, and Antonemkov (56) in a Soxhlet laboratory extraction unit with benzene and 12-, 25-, and 50-mesh soybeans. As would be expected, increasing amounts of oil were extracted in a given time as the particle size was decreased.

Certain practical considerations make the large scale extraction of powdered soybeans difficult. The powdered material, unless agitated, is not readily wetted by the solvent. Even after wetting the bean flour would need to be continuously agitated to secure adequate extraction. Counter-current movement of the solvent through the flour would be difficult to secure and would require a high ratio of solvent to solids. After extraction had taken place, removal of the solvent-oil mixture from the flour would be troublesome. Volatilizing the solvent from the flour would be more difficult than from a material forming a more porous mass, and it is probable that a considerable amount of the fine flour would be carried over with the solvent vapors to the condensers, fouling them and contaminating the liquid solvent.

In practice, flakes not more than one-hundredth of an inch thick have been found to be the most satisfactory form in which to place the soybeans for solvent extraction. Flakes not only present a large surface and a small penetration distance, but allow the solvent to flow readily without carrying excessive amounts of fine material.

Desirable Flaking Conditions

Soybeans are commonly flaked by passing them between a pair of smooth rolls. The proper diameter of the rolls is a matter on which there is some difference of opinion. Although beans have been flaked experimentally in the present work on rolls as small as 10 inches in diameter, rolls are used in industry up to at least 48 inches in diameter. The conventional formulas used in calculating diameters for roll crushers do not apply to flaking rolls.

Work by Yates (55) in this laboratory led to the conclusion that the optimum conditions for flaking soybeans are as follows:

(1) a moisture content in the beans of 10 to 12 per cent; (2) tempering prior to flaking; (3) cracking the beans before flaking; (4) flaking on hot rolls; and (5) the use of rolls larger than 16 inches in diameter. Succeeding work confirms the need for 10 per cent or more of moisture in the beans for good flaking. Very dry beans may be moistened with steam prior to flaking.

Tempering the beans consists in heating them either by dry heat or direct steam to a temperature between 65 and 85°C. (149° to 185°F.). The purpose of heating is to render the beans more plastic and decrease the production of undesirable amounts of fine materials in the flaking process. Unless the beans are below 10 per cent moisture content, dry heating is preferable to steaming. It is not necessary to heat the rolls as they become warm enough in operation to give good flaking.

Cracking the beans prior to flaking is desirable. Cracked beans may be flaked on smaller rolls than whole beans, partly because of the smaller size and partly because of the greater coefficient of friction. Rolls as large as 48 inches in diameter are probably necessary for whole beans while beans cracked into 6 to 8 pieces have been successfully flaked in this laboratory on 18-inch rolls. Large sized flakes, such as those obtained from whole beans, when dry tend to break into more fines than flakes which are initially smaller. If the large flakes are rather moist they may curl excessively, causing undesirable bulking with consequent reduction in capacity of the extractor.

Flaking Methods

Cracking beans for flaking is commonly done between corrugated rolls, preferably with LePage corrugations*. Beans have also been cracked in this laboratory on smooth rolls but smooth rolls are not satisfactory for beans containing much moisture or for steamed beans. Rolls with ordinary longitudinal corrugations of the V type have been used satisfactorily in this work. However, most of the beans used in the laboratory were cracked in an attrition type of laboratory mill having a stationary and a revolving grinding plate. This was mounted directly above and discharged by gravity into the flaking rolls. The beans were fed by gravity into the cracking mill. Tempering was done prior to

* A type of corrugation patented by Jules LePage, U. S. Pat. 1,183,573 and 1,183,574, May 16, 1916. Designed to cut with the production of a minimum of fine material. The corrugations are U- rather than V-shaped. The corrugations on one roll of a pair are longitudinal and the other spiral.

cracking by one of two methods: by blowing steam from a pipe attached to a hose directly into the beans or by heating in a tunnel drier. Beans tempered by the latter method were cracked more readily and at a higher rate in the mill than were the steamed beans.

Flaking for the earlier studies was largely done on a set of 12-inch rolls after cracking in an 8-inch roll crusher. In most of the later work the cracking was done in the mill described above and flaking by a pair of 18-inch diameter 8-inch face rolls designed originally as an oat crusher. These rolls operated at 200 r.p.m. and had a capacity of about 150 pounds of beans per hour. Thickness of the flakes changed with variations in beans, rate of feeding, and adjustment, with a usual maximum of 0.01 inch.

Studies made on a flaker patented by Truax (49) were only moderately successful, the initial flaking rate of 125 pounds per hour dropping to 80 in less than an hour. Succeeding runs showed a similar decrease in capacity, caused apparently by the rolls becoming hot. This reduction in capacity as the rolls become hot during operation occurs frequently. A pair of 10-inch rolls operated satisfactorily for 18 hours after which the capacity was so reduced that it was necessary to cool them before further use. This difficulty was not experienced with the 18-inch rolls which, because of their construction, did not become as hot during operation. These rolls were not of solid construction but consisted of a ½-inch thick rim attached to a shaft by an integral cast spider.

THE FIRST LABORATORY PILOT PLANT UNIT

Preliminary Laboratory Studies

In the solvent extraction of any oil-bearing seed, it is not merely necessary to bring the solvent into contact with the seed, but the solvent must be brought into contact with the oil in the seed. It has been noted that for efficient extraction it is necessary to roll the beans into flakes. This flaking process loosens the hull, which the solvent penetrates with difficulty, and reduces the distance the solvent must travel. The oil in the bean is contained in cells having definite but thin walls. Some of these cell walls are broken in flaking allowing easier access by the solvent to the oil.

It has been assumed that the solvent reaches the center of the flakes by diffusion. Once the solvent reaches the oil, the oil dissolves. There is then set up a concentration gradient between

the oil dissolved in the solvent in the flake and the oil-free solvent outside the flake. As a result, oil and solvent tend by diffusion to equalize the concentration of oil in the solvent. It seems evident that the rate of diffusion of the oil outward and of the solvent inward is a function of the concentration difference and an inverse function of the distance. This is expressed by Fick's law as follows:

$$ds = -DA \frac{dc}{dx} dt$$

where s = quantity of solute, D = a coefficient, A = area over which diffusion occurs, x = distance, t= time, and dc = change in concentration. This law assumes diffusion to be occurring in a porous body. It is assumed that the resistance to diffusion of oil from the center of a porous body into the solvent surrounding it may be divided into two parts: the resistance to diffusion through the capillary structure of the body, and the resistance to diffusion into the solvent outside the body.

The mechanism of solvent extraction of soybean oil has been studied as a problem in extraction from a porous mass by Boucher, Brier, and Osburn (6). Most of their work involved a study of the extraction of soybean oil from porous clay plates by perchloroethylene and a carbon tetrachloride-ethylene dichloride mixture. They concluded that the extraction process was one of pure molecular diffusion, with diffusion coefficients which were practically constant for any one temperature and system and independent of liquid concentration. Similar work by King, Katz, and Brier (26), with trichloroethylene as a solvent, gave similar results. Extraction of soybean flakes with trichloroethylene gave data resembling that obtained with the porous clay plates. When the logarithms of the fraction of extractible oil remaining in the soybean flakes were plotted against extraction time, they gave curved lines as compared with straight lines characteristic of the appropriate theoretical diffusion equation from the porous plate data. This indicates that while the soybean flakes are not uniform porous bodies with uniform oil distribution, yet in general the extraction of the flakes is a diffusion process similar to that obtained with porous bodies.

To secure data on several factors involved in the extraction of soybean oil with trichloroethylene prior to designing larger extraction apparatus, small batch extractions were made in this

laboratory by Measamer (37). Weighed samples of flaked soybeans were placed, together with measured amounts of trichloroethylene, in a wide-mouthed bottle. Here they were stirred for a given length of time after which the miscella was filtered from the flakes in a covered funnel. The specific gravity of the miscella was determined by a Westphal balance, and the amount of oil extracted was calculated from the miscella gravity and the amount of solvent used. A typical curve obtained for flakes containing 10 per cent moisture and showing the relationship between oil extraction and time of extraction is shown as (A) in Fig. 3. These data (Table 6) substantiate the diffusion theory of extraction since there is a straight line relationship between the logarithm of the time and the per cent oil extracted. Similar data were secured for dry flakes and are plotted as curve (B) in Fig. 3. The fact that the curves plotted from these data are practically identical indicates that moisture variations from zero to 10 per cent have very little effect on the rate of extraction.

Other work in this laboratory showed the same oil extraction after one hour in a Sohxlet extractor for beans with 5.63 per cent and 9.03 per cent moisture. Increasing the moisture content to 28 per cent showed a gradual decrease in the amount of oil extracted. The higher moisture contents were secured by steaming the flakes and allowing them to stand 24 hours before extracting. It is possible that the distribution of moisture in the flakes may have been different in the steamed flakes than in the unsteamed ones. Swelling of the protein in the flake due to a high moisture content may also retard oil extraction.

Data were also secured on the amount of oil extraction in various lengths of time with no agitation, mild agitation, and rapid agitation, using a solvent-bean ratio of 4.5 to 1.0 (by weight).

TABLE 6

RELATION OF AMOUNT OF OIL EXTRACTED TO EXTRACTION TIME

Time of contact in minutes	Grams of oil extracted per 100 gr. beans
1	
3	9.2
6	13.2
9	16.0
	17.5

Flakes 0.025 in. thick; 25°C.; 450 g. solvent, 100 g. beans; 10 per cent moisture in beans.

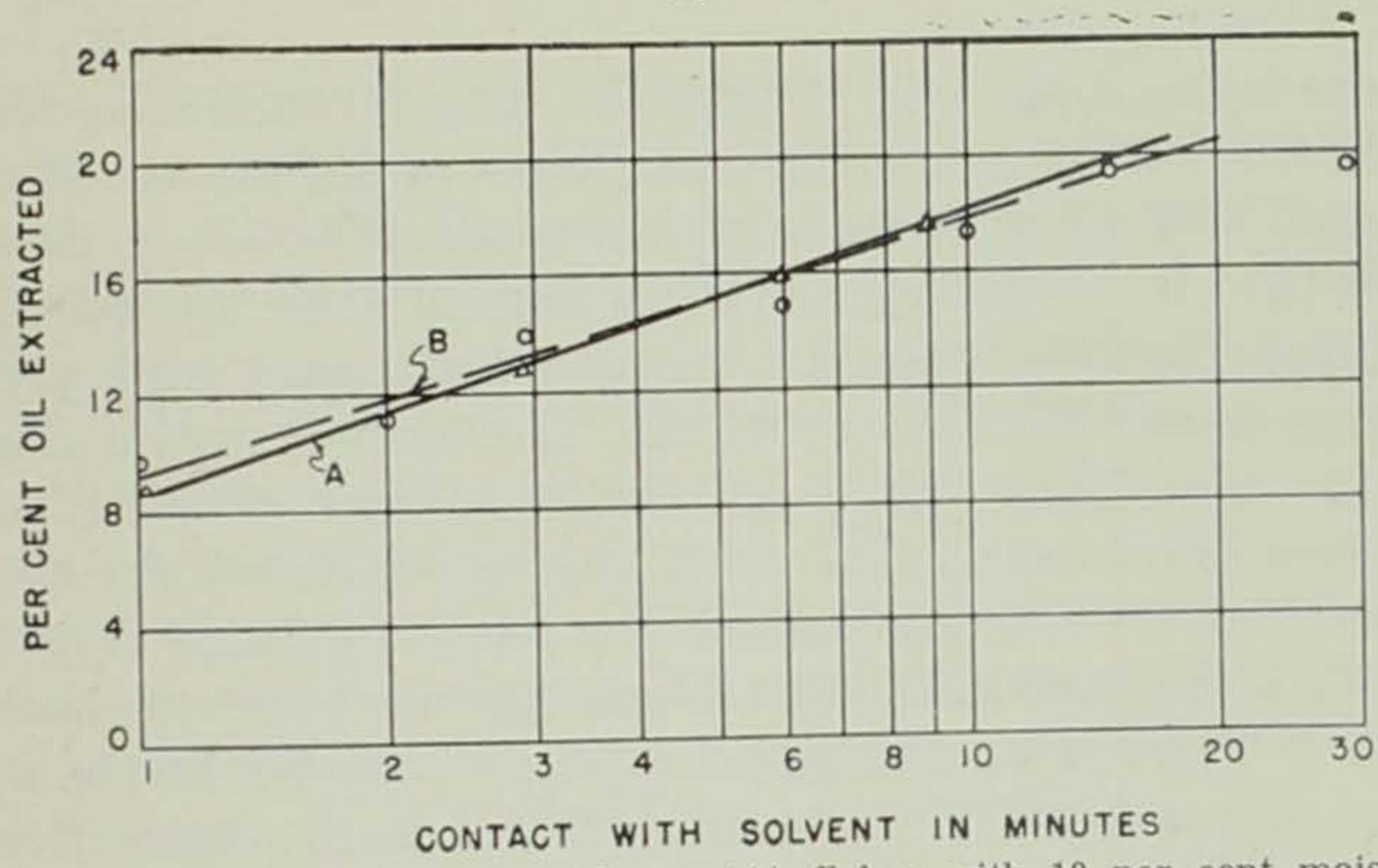

Fig. 3. Amount of oil extracted from—(A) flakes with 10 per cent moisture, (B) dry flakes.

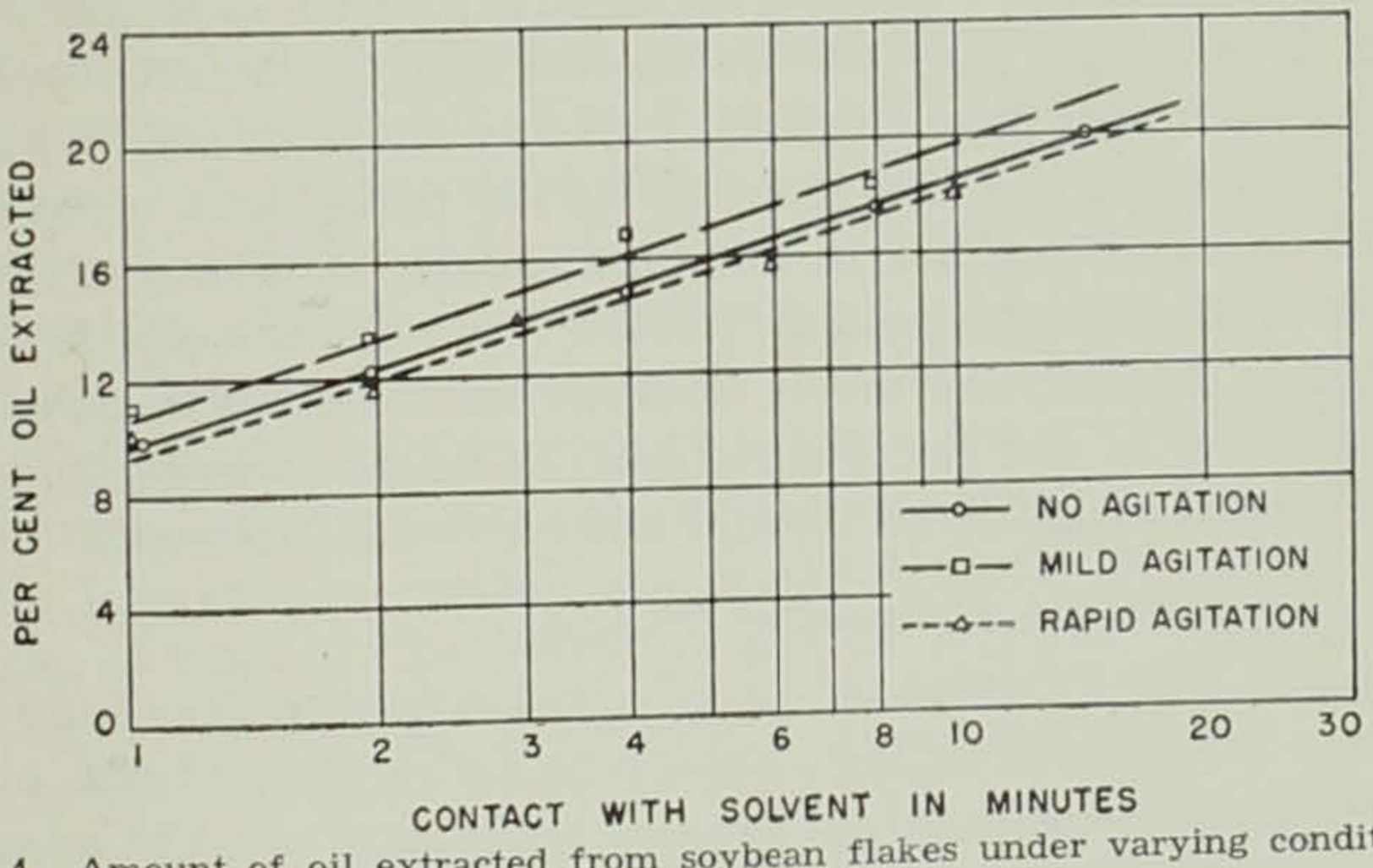

Fig. 4. Amount of oil extracted from soybean flakes under varying conditions of agitation.

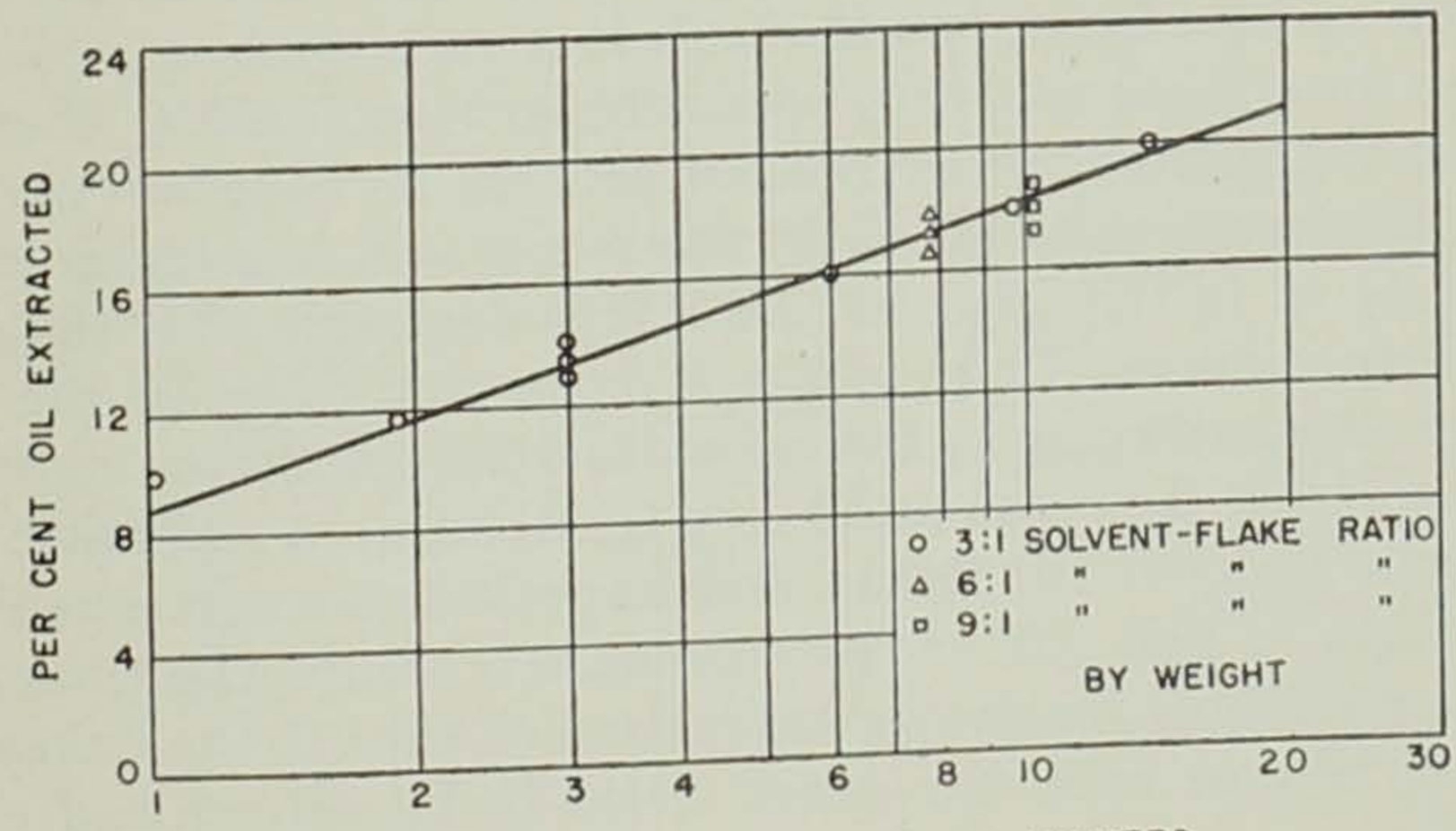

Fig. 5. Amount of oil extracted from soybean flakes with different solvent-flake ratios.

The semi-logarithmic curves (Fig. 4) are approximately identical straight lines, thus indicating no significant differences in extraction rates. This agrees with the conclusions of Boucher, Brier, and Osburn (6) on the extraction of oil from a porous solid where it was found that the external surface resistance was negligible compared to the internal resistance to diffusion, thus making the latter the controlling factor in extraction time.

When extraction rates using solvent-bean weight ratios of 3, 6, and 9 to 1 were determined, the resulting data, (Fig. 5), indicate no significant difference in extraction rates with the different ratios. Figures 4 and 5 taken together confirm the theory that internal diffusion is the controlling factor in the extraction rate.

As would be expected, increase in temperature increases the extraction rate. An increase from 25°C. to 60°C. showed an increase of about 20 per cent in the amount extracted for a given extraction time. Flake thickness affects the extraction rate and a decrease in thickness from 0.025 to 0.015 inch doubled the amount of oil extracted in one minute. In 10 minutes about 25 per cent more oil had been extracted from the thinner than from the thicker flakes. The effect of flake thickness was observed in the operation of the regular extraction units—where it was concluded that the thickness should not exceed 0.010 inch.

Development of the Extractor Design

In the development of an efficient solvent extractor certain factors need consideration. Complete extraction of the oil by a single bath of solvent would require an infinite amount of solvent. Extraction in this manner even to a reasonable amount of residual oil, such as 1 per cent, would result in a very dilute miscella thus causing the solvent recovery operation to be expensive. Therefore, it is desirable that the extraction process be counter-current to secure minimum residual oil in the flakes and maximum concentration of oil in the miscella. Also, it is desirable that the operation be continuous. This makes it necessary to move the flakes through the extractor in the opposite direction to the movement of the solvent. It is also necessary to maintain a sufficient depth of solvent to cover the flakes, and to provide some mechanism to move them in and out of the solvent. To secure the time in the solvent necessary for proper extraction of the oil, the flakes must move at a slow rate, the exact speed to be determined experimentally.

In preliminary stages of the experimental work it appeared that a screw conveyor might be used for the movement of the flaked beans through the extractor, and experiments were begun with a small steel and glass extractor utilizing such a conveyor.

This extractor consisted of a 4 in. steel pipe approximately 4 ft. long attached at a 60° angle to a 3 ft. length of 4 in. glass tubing, the latter containing a screw conveyor. The glass tube was set vertically and filled with trichloroethylene. Flaked beans were introduced into the bottom of the glass tube by means of a screw operating in the inclined pipe. The preliminary work showed that a screw conveyor would carry the flakes satisfactorily through the solvent in both the inclined and vertical tubes.

Since the flakes floated readily in the trichloroethylene it was thought possible that they could be conveyed down the side tube into the bottom of the vertical tube, and allowed to rise to the top, where they would be picked up by a short section of conveyor and elevated to a discharge point above the solvent level. It was found that while the flakes and hulls would float in pure trichloroethylene any hulls which had separated would not float in the solvent containing as low as 3 per cent oil, since the addition of the oil to the solvent produced a mixture having a lower gravity than the pure solvent. As a result many of the bean hulls settled out at the bottom. It was not considered practical to remove these hulls from the bottom of the extractor nor to separate them from the beans before or during the flaking, so that this method was abandoned in favor of positive feed of the flakes by a screw.

When removing the miscella at the bottom of the glass tube,

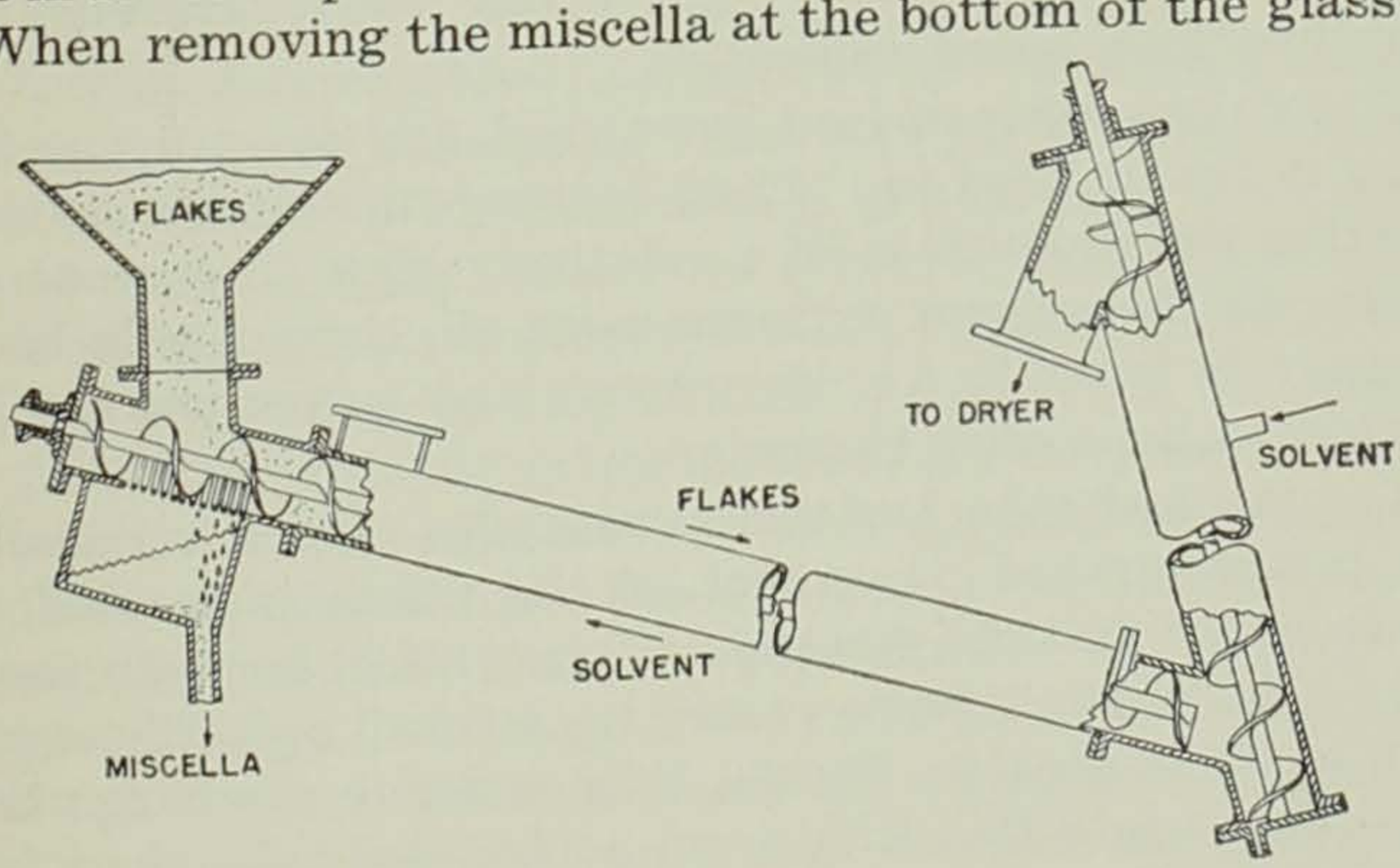

Fig. 6. The first pilot plant extractor.

it was found impossible to secure a concentration of oil at that point greater than 4.8 per cent. However, as much as 50 per cent oil was secured at the upper end of the inclined tube. This indicated that extraction was largely taking place in the inclined tube, a negligible amount occurring in the vertical tube. This resulted from the fact that the solvent-oil mixture decreased in density with increase in oil content, the mixture rich in oil thus rising readily up the inclined tube. Although the movement of the solvent was downward in the vertical tube, the solvent became lighter as it dissolved more oil. This decrease in gravity tended to make it rise, thus producing eddy currents and mixing the solvent through the tube, preventing any build-up of oil content.

After adding a slotted miscella outlet at the upper end of the inclined tube, above the point at which the flakes were fed in, and using an extraction time of 45 minutes, with a miscella containing 27.4 per cent oil, it was found possible to reduce the oil content of the flakes below 2 per cent. The results were considered promising enough to warrant the design of a larger unit.

The Pilot Plant Extractor

The extractor which was designed as a result of the previous experiment is shown in Fig. 6. The extractor was made of 6-inch pipe, the inclined leg being 25 feet long with 22 feet of actual extraction length. The riser leg was 13½ feet long with 7 feet of active length. Both the inclined or extractor leg, and the riser leg, were fitted with solid flight screw conveyors electrically driven through a speed reducer.

The flakes were carried downward and the solvent flowed upward in the inclined leg, giving counter-current extraction and permitting the production of a relatively high oil content in the miscella. As the solvent extracts more oil, the miscella becomes progressively lower in specific gravity and moves upward in the extractor without eddy currents.

The inclined tube has two advantages over a vertical tube. First, better upward movement of the flakes is secured at an angle from the vertical, since the friction effect between the meal and the sides of the tube necessary to cause an upward movement by the screw is greater. Second, it is easier to discharge the extracted flakes into the drier from an inclined tube than from a vertical tube.

Two openings were provided at the upper end of the extractor tube for feeding in the flake, one above and one below the miscella outlet. Both were on the upper side of the tube and were flanged for the attachment of the feeding hopper. It was found that the flakes could be fed at either point but that the upper position was more satisfactory. The buoyancy of the flakes in the miscella made it difficult to feed them regularly into the lower opening. When feeding into the upper opening, the rate of feed could be changed by changing the diameter of the conveyor shaft at the point where the flakes entered. This was found necessary in order to prevent a pumping action due to the resistance to flow of the solvent through the flakes, which caused the solvent inlet level to be high. Data pertaining to the extractor and its performance are given in Tables 7 and 8. The data in Table 8 may be used to show that the counter-current continuous extraction is practically logarithmic, as would be expected.

TABLE 7

OPERATING DATA ON EXTRACTOR OF FIG. 6.

Speed of extractor conveyor (inclined leg)	1.33 R.P.M.
Capacity	105 lb. per hr.
Capacity, shaft diameter increased	85 lb. per hr.
Capacity per cu, ft. of free extractor space	15 lb. per hr.
Extraction time	About 45 min.
Miscella range	12 to 28 per cent
Suspended solids in miscella leaving the extractor	3 to 5 per cent
Solvent in flakes leaving extractor	90 lb. per 100 lb. of flakes

Drying the Meal

In any system of solvent extraction the extracted material leaves the extractor containing some solvent. It is necessary to remove this solvent to produce a meal suitable for feeding purposes and to recover the solvent for reuse. It was found that the output from the extractor averaged 90 lb. of solvent and 17 lb. of water (corresponding to 12 per cent moisture in the original flakes) for each 100 lb. of extracted meal after drying. It is common practice to remove the solvent from the meal by heating the latter in steam-jacketed tubes through which it is moved by screw conveyors. This process is spoken of commonly as "drying" and the tubes as "driers", although if the concept of drying is that of the removal of water this designation is a misnomer. The primary purpose of the drier is the removal of solvent, the removal of water being largely incidental to the solvent material.

TABLE 8
OIL CONTENT AT VARIOUS POINTS IN THE EXTRACTOR (FIG. 6).

Distance from miscella outlet in feet	Per cent oil in miscella
0	31.5
6	16.0
10	10.0
14	6.0
18	3.0
22	1.5
29	0

The driers used on the first pilot plant unit were designed on the basis of such data as were available in the literature, and from that secured in the preliminary studies. Essentially they consisted of three 6-in. steel tubes jacketed by 8-in. pipes and fitted with screw conveyors. Details are shown in Fig. 7. To give a greater mixing action than would be secured by the use of the conventional spiral-ribbon conveyor, a conveyor was made using a flighting of $\frac{3}{8}$ inch steel rod formed into a continuous spiral and attached to the central shaft by means of radial rods. To secure a further mixing effect auxiliary flighting of 18-in. pitch was added to the first and third drier tubes. Horizontal connecting bars were added between spirals in the second drier. Modifications of these conveyors were subsequently made to improve performance and to prevent plugging of the tubes. These modifications took the form of replacing some of the steel rod spiral with a solid flight conveyor screw.

Provision was made for the introduction of steam into the lower drier through nozzles on the lower side of the tube. The purpose of the steam was to sweep out the last traces of solvent from the meal. Steam was used for this purpose instead of air since it could be condensed with the solvent vapor in the con-

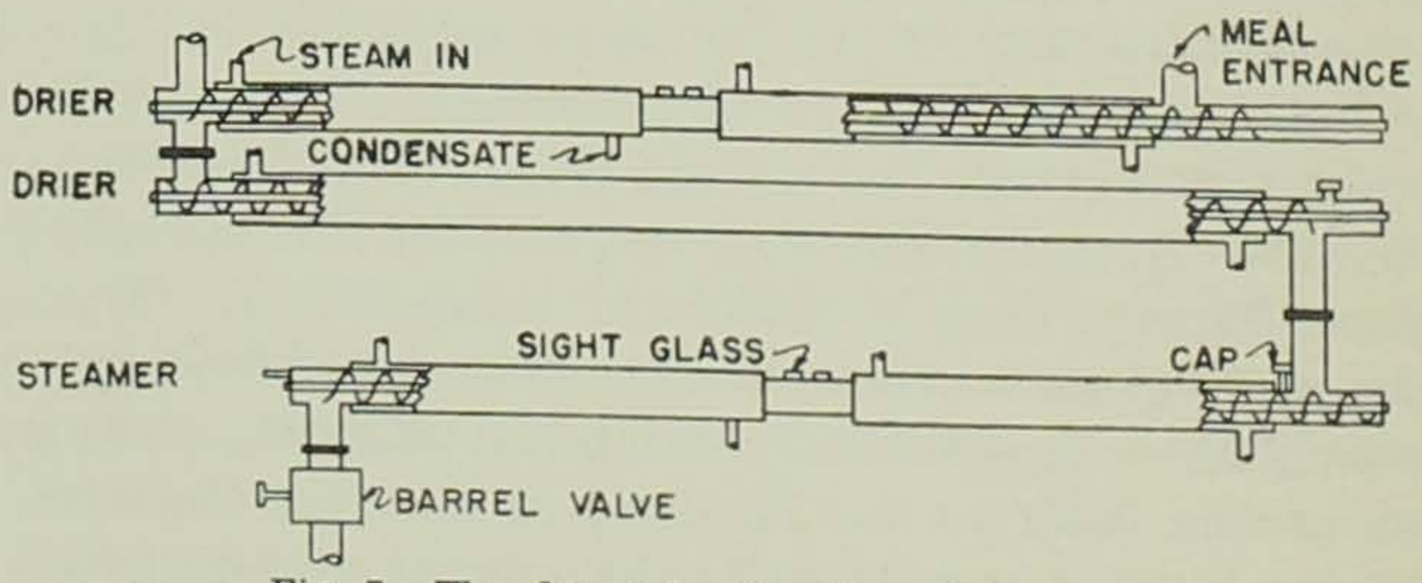

Fig. 7. The driers for the first pilot plant.

denser whereas air or other noncondensable gases introduced into the condenser would have to be vented to a recovery system to remove the solvent vapor carried by it. Because of clogging of the steam nozzles and the tendency of the steam, entering at high velocity at the bottom of the drier, to blow fine meal into the condenser the bottom nozzles were abandoned in favor of a single nozzle at the discharge end. The meal was removed from the bottom drier through a barrel valve.

Originally the vapors from the driers were taken off through two vapor lines—one from the discharge of drier No. 1 and the other in the transition between driers No. 2 and No. 3. Runs were made using each of these separately and using a third line connected to the center of No. 1. These lines carried the vapors to a vertical condenser above the driers. Because of the small size of the vapor lines (2 or 3 in. pipes), and the necessity of blowing in considerable steam to raise the heavy solvent vapor to the condenser above the driers, considerable fine meal was carried to the condenser. It was found that much more satisfactory operation could be secured by lowering the condenser into the same plane as No. 2 drier. Here it was set horizontally and connected to the discharge end of No. 1 drier by a 5 in. vapor line so that the heavy vapor flowed into the condenser by gravity. The condenser was operated with water on the shell side and vapor in the tubes.

A separator to separate the solvent and water was connected below the condenser. This was a piece of 6 in. pipe, closed at both ends, mounted vertically and with a rectangular sight glass in the side. The solvent-water mixture flowed into the side at about the solvent and water interface, and the solvent was removed from the bottom through a U-tube to maintain the proper level, and pumped to the extractor. The solvent flow rate into the extractor was metered by a rotameter. The water overflowed to the sewer through an opening near the top of the separator.

Data on the driers used with this pilot plant, and their operation, is summarized in Table 9.

Stripping the Solvent from the Oil

The concentration of oil in the miscella as it comes from the extractor may vary considerably depending upon the type of extractor, the solvent used, and the extraction conditions. In the present situation using trichloroethylene to extract soybean oil,

TABLE 9
DRIER DATA

Composition of meal:

	Water*	Oil*	Solvent*
Entering upper drier (No. 1)	17.0	1.25	90.0
Leaving drier	8.5	1.25	9.0

Temperature:
Entering 25°C. (77°F.)
Leaving 100°C. (212°F.)
Solvent vaporized in upper driers — 72 lb. per 100 lb. meal
Water vaporized in drier — 8.5 lb. per 100 lb. meal

Thermal data:
Trichloroethylene
Boiling point at 760 mm. pressure — 86.7°C. (186.4°F.)
Specific heat at 20C. (liquid) — 0.223 B.t.u. per lb. per °F.
Latent heat of vaporization (at B.P.) — 103.1 B.t.u. per lb.
Steam distillation temperature — 73°C. (163.4°F.)
Soybean oil
Mean specific heat (25-100°C.) — 0.5 B.t.u. per lb. per °F.
Nonvolatile at temperatures used
Soybean meal
Mean specific heat (25-100°C). — 0.4 B.t.u. per lb. per °F.
Heat transfer coefficients:
Steam temperature in jacket — 45 to 50 lb. per sq. in. gauge
Meal, total free volume of tube — 25 to 30%
Overall heat transfer coefficient — 3 to 4 B.t.u. per hr. per sq. ft. per °F (based on total inside heating area).
Capacity of drier:
Wet meal (No. 1 drier) — 6 lb. dry meal per cu. ft. free volume
Dry meal (No. 2 drier) — 8 lb. dry meal per cu. ft. free volume
Linear vapor velocity — Not over 2 ft. per sec.

Composition of meal leaving No. 2 drier (entering lower drier)
Water — 3 to 4 lb.
Oil — 1.25 lb. per 100 lb. dry meal
Temperature of meal in lower drier: 115-118°C. (240°F.)

Solvent and water evaporated:
Trichloroethylene — 8 lb. per 100 lb. meal
Water — 5.5 lb. per 100 lb. meal
Heat transfer coefficient in lower drier
Meal — 20 per cent of total free volume, 1 B.t.u. per hr. per sq. ft. per °F.
Capacity of lower dried — 10 lb. dry meal per cu. ft.

* Lb. per 100 lb. of meal.

it was found that under average conditions of operation a miscella containing approximately 20 per cent oil would be produced. Thus for each pound of oil processed it is necessary to recover from it four pounds of solvent. Although pure trichloroethylene boils at 86.7°C. (188°F.) it cannot be vaporized from the miscella at that temperature. Thus it was desirable to determine the boiling points of trichloroethylene-soybean oil mixtures of various proportions and under various pressures.

To determine the boiling point of the miscella at reduced pressures the equipment shown in Fig. 8 was used by Measamer (37). The flask was connected to the condenser through a section of rubber tubing in such a manner that the weight of the miscella in the flask could be determined without disconnecting the flask. Miscella of known concentration was placed in the flask

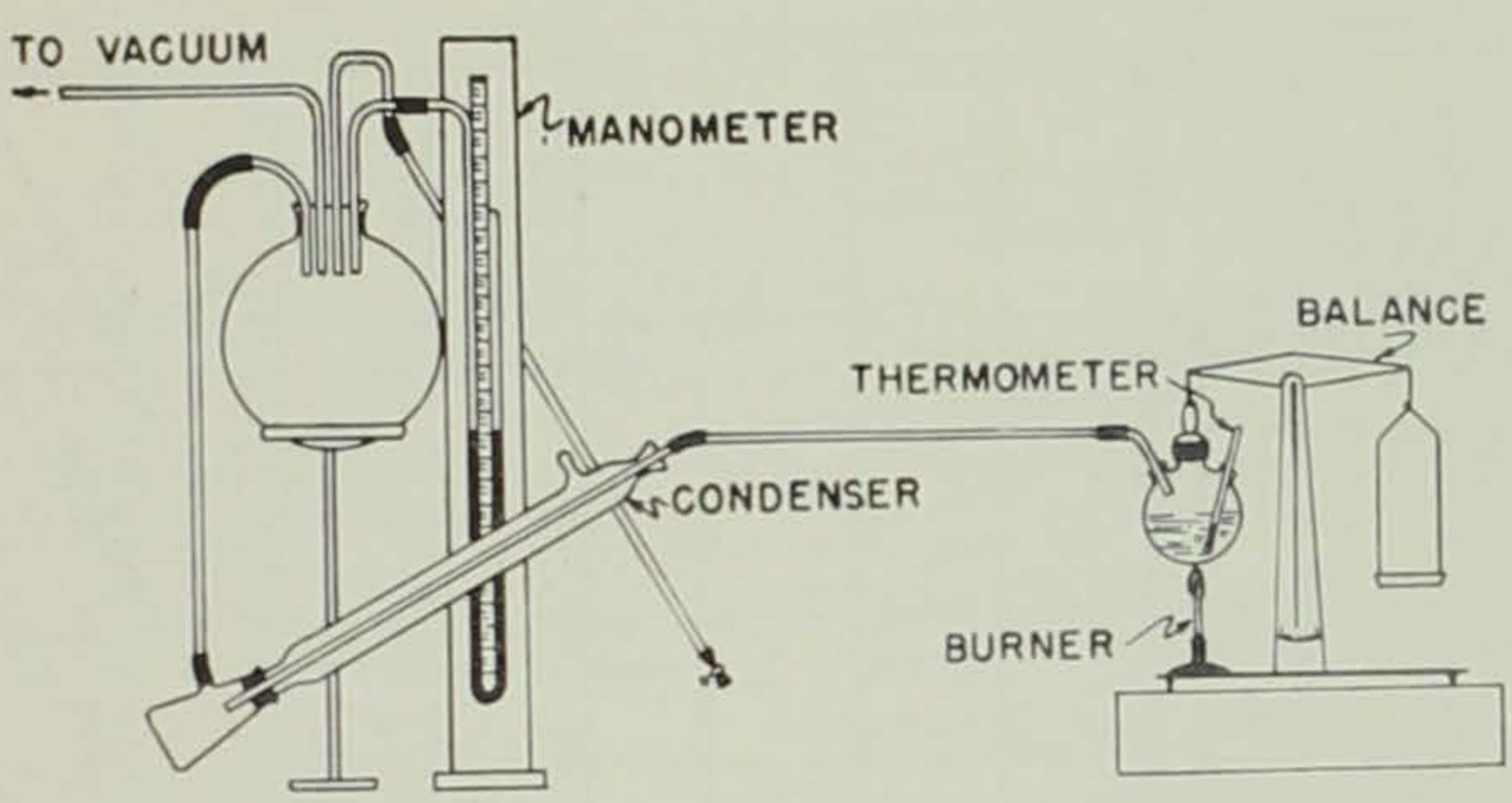

Fig. 8. Apparatus for determining the boiling point of trichloroethylene-soybean oil mixtures.

and balanced by weights on the right pan. The pressure on the condenser was adjusted to the desired value and held constant during the run.

A ten gram weight was removed from the pan and heat applied to the flask. At the point when the flask and its contents just balanced the weights a temperature reading was made and the balancing weight reduced by 10 grams. This procedure was repeated until the temperature reached 120°C. (248°F.). The pressure was next changed to a new value and a new series of temperatures recorded. The data are shown in Fig. 9 as the relation between the percentage of trichloroethylene in the miscella and the boiling temperatures, for various pressures. In Fig. 10 the vapor pressures for various concentrations at certain temperatures are shown.

Boiling point data were also obtained for solutions at atmospheric pressure (774 mm of mercury) by other investigators in this laboratory using two different methods. First a three-necked 500-cc flask was fitted with a reflux condenser and a thermometer. About 200 cc of solutions of known concentrations of oil and trichloroethylene were made up and boiled until a constant temperature was reached. Each solution was allowed to cool and the specific gravity determined to check the concentration. For the second method the flask was fitted for straight distillation. Exactly 300 cc of solution containing 19.5 per cent oil by weight was placed in the flask and boiled. The temperature was noted each time the volume of distillate collected passed a 10 cc mark on a graduated cylinder. The percentage of oil in the flask at any temperature could thus be calculated.

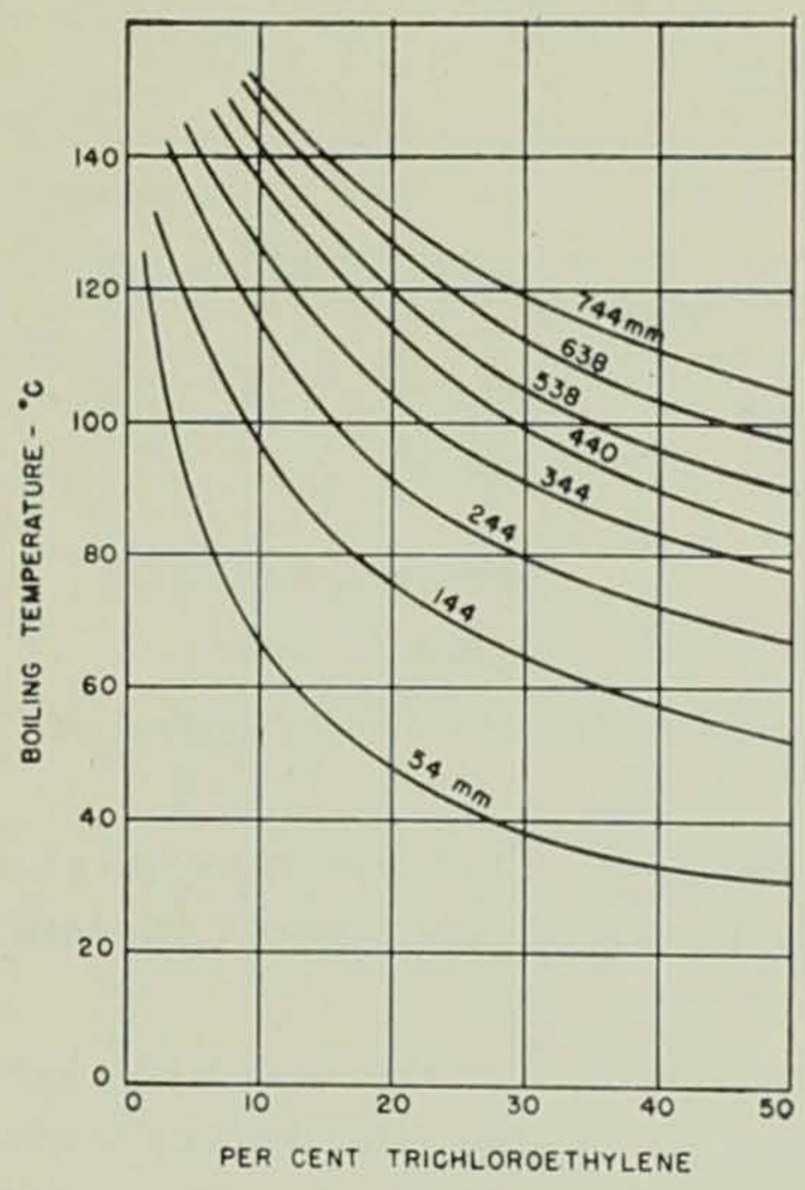

Fig. 9. Boiling points of trichloroethylene-soybean oil mixtures at various pressures.

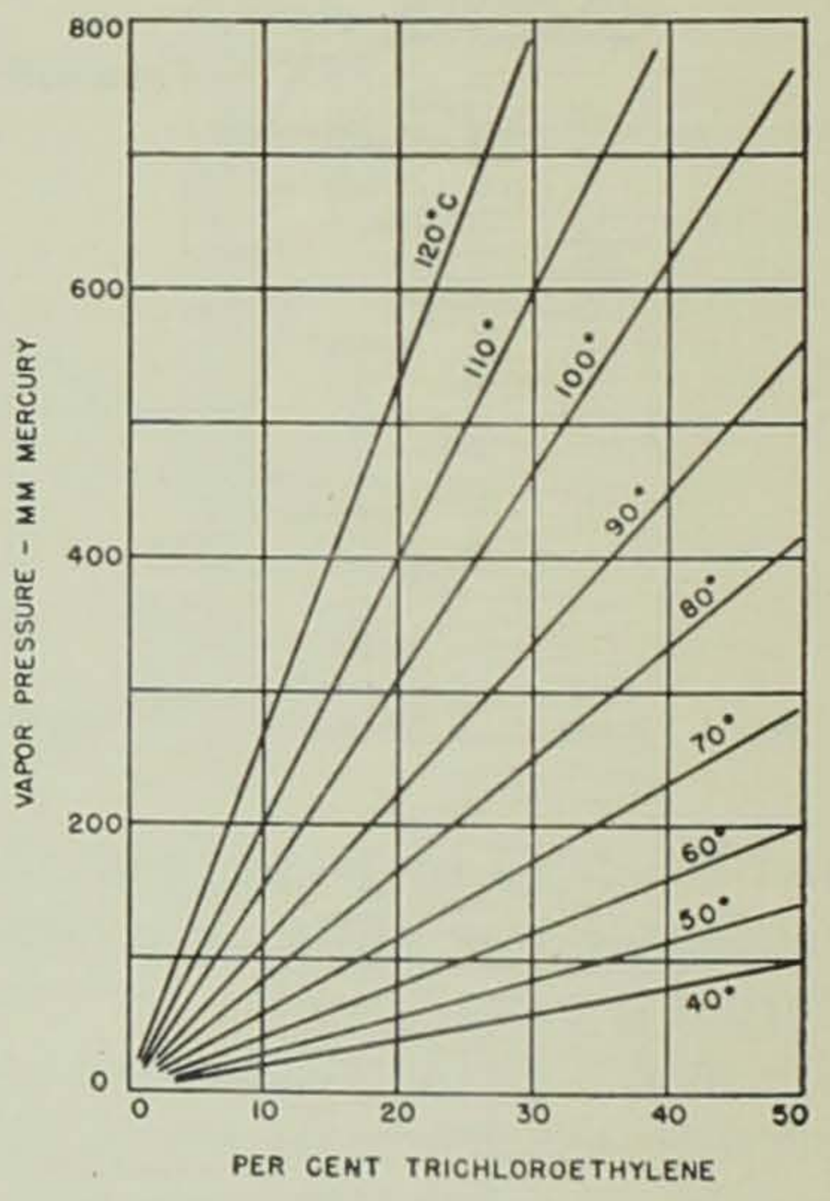

Fig. 10. Vapor pressures of trichloroethylene-soybean oil mixtures at various temperatures.

The data obtained by the two methods (Fig. 11) agree very well for amounts of oil below 80 per cent. Above this point the results are not in close agreement for several reasons. When refluxing, some solvent is held up in the condenser making the boiling solution richer in oil than before or after boiling; the smaller the total amount of solvent the greater the percentage error. When the distillation method was used the temperature was rising so rapidly at the end that the time of definite boiling points could only be estimated. From a practical standpoint, the exact boiling points of solutions containing more than 80 per cent oil are of little value, since it is not possible to heat the miscella to such high temperatures without damage to the soybean oil.

From the data on boiling points it seems evident that it is not practical to remove completely the solvent from the oil by boiling, even under vacuum, at a temperature below 120°C. (248°F.). It is desirable to keep the oil temperature below this point to prevent darkening of the oil. Since it seemed impractical to completely remove the solvent by simple boiling, the next logical step was to investigate conventional stripping methods using steam or an inert gas.

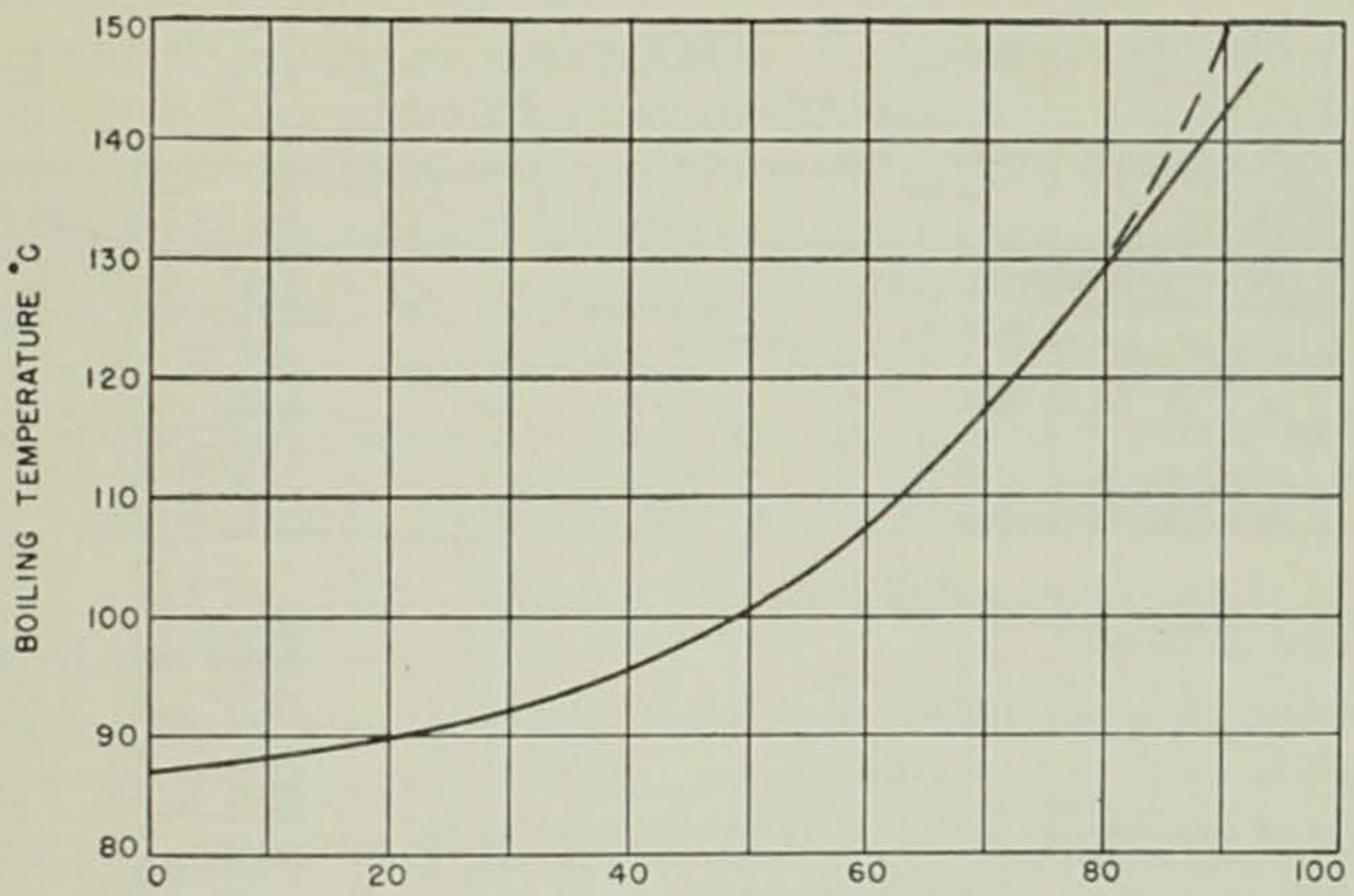

Fig. 11. Boiling points of trichloroethylene-soybean oil mixtures at atmospheric pressure.

The Pilot Plant Stripper

The original pilot-plant stripping equipment was made in two units: The miscella preheater and the stripping still. The essential part of each unit was a tube section consisting of 19 brass tubes ¾ in. O.D., No. 20 B.W.G., and 5 feet long, mounted in a shell of 5-inch standard pipe. Steam at 15 and 25 pounds per square inch was used on the shell side, the miscella filming down the inside of the tubes. Each tube section was fitted with a jacketed kettle on the bottom and with a miscella inlet and vapor outlet at the top, with the outlet connected to a condenser. The miscella ran from the preheater through a pipe to the stripping still, and steam was blown in through the bottom of the stripper to strip out the last traces of the solvent.

In the first run it was observed that the miscella from the extractor contained some fine particles of meal in suspension. An 8-mesh screen was then placed in the miscella outlet housing but some fine material passed through the screen. Operation during the first part of a run was satisfactory but after a time the accumulation of fine material in the bottom of the stripper began to cause foaming of the oil. The oil leaving the stripper was dark in color and contained suspended solids. Eventually the flow of oil became very erratic due to the accumulation of solids. Bumping and foaming increased until solids were actually thrown into the vapor line.

TABLE 10
OPERATING DATA — RUN 20

Length of run	101 hr., 8 min.
Weight of flakes fed to extractor	8,350 lb.
Flakes fed to extractor per hour (average)	82.56 lb.
Moisture content of flakes (average) wet basis	8.5%
Oil content of flakes (average) dry basis	21.84%
Weight of meal from extractor	5,904 lb.
Moisture content of meal (average)	2%
Weight of meal from extractor (dry basis)	5,786 lb.
Oil content of meal from extractor (average) dry basis	1%
Weight of oil in extracted meal	57.86 lb.
Weight of oil from extractor (total)	1,553 lb.
Weight of sludge from filters (total)	394 lb.
Volatiles in sludge	55.4%
Oil content of non-volatile sludge	31.2%
Weight of dry solids in sludge	175.7 lb.
Weight of oil in sludge	54.8 lb.
Weight of dry fat-free solids in sludge	120.9 lb.
Weight of dry fat-free solids in sludge corrected to 8.5 per cent moisture (wet basis) and 21.84 per cent oil (dry basis)	155.33 lb.
Per cent of original feed reaching filter	1.86%
Condenser water used per hour	990 lb.
Temperature of condenser water	11°C. in, 30°C. out
Total power consumed (extractor)	55 KWH.
Power per ton of beans processed	13.1 KWH.
Steam consumed per pound of flakes	1 lb.

These conditions necessitated a redesign of the apparatus. To remove the fines in the miscella two bag filters were constructed to replace the previously-used settling chamber. The filter bag of 8-ounce canvas was held in a 2½ foot length of 8-inch pipe and supported by a coarse screen. The miscella flowed by gravity from the extractor under an eleven-foot head through the bag filter to an enclosed storage tank from which it was pumped to the stripper. The bag filters were arranged so that one filter was used at a time.

The stripper was reconstructed by replacement of the jacketed kettle at the bottom of the tubes with an open bucket. The miscella was fed into the top of the preheater and filmed down through the stripper tubes into the bucket forming a liquid seal. Any solid material washed into the bucket where it could be removed without interfering with the stripping operation.

General Operation of the Unit

After the above design change the unit operated well, extracting beans at the rate of about 85 pounds per hour to a 1 per cent oil content, with solvent losses held down to 1 per cent of the weight of the processed beans. The oil was apparently free from solvent. Later work showed that this was not true, although

under good stripping conditions the oil probably contained not more than 0.5 per cent solvent. With the exception of the miscella pump and the solvent pump, each of which was driven by a ¼ H.P. motor, the whole unit was driven by a 5 H.P. electric motor through a speed reducer with power from the reducer carried by roller chain drives. Detailed data is given in Table 10 for the type of operation obtained at the end of the development period.

MODIFIED EXTRACTION SYSTEM WITH INTEGRAL STRIPPER

Design of the Unit

While the large-scale unit described in the preceding section operated well, it was believed that it could be simplified to make the cost of equipment lower, and the operation more easily handled. With this in mind a unit was designed in which the stripper was built as an extension on the extractor, thus eliminating the necessity of a pump and flow meter. This unit is shown in Fig. 12.

The stripper consisted of a sheet metal box built on the top of an extension of the extractor tube, the bottom of which was provided with a series of inlets for stripping steam. Twenty-four ⅛-in. pipe nipples with pet cocks were used to admit the steam. Several other devices, such as porous distributing plates and special nozzles were tried to permit steam to be blown into the miscella, but none could operate without becoming clogged.

Operation of the Unit

In addition to short preliminary runs, two runs of four days

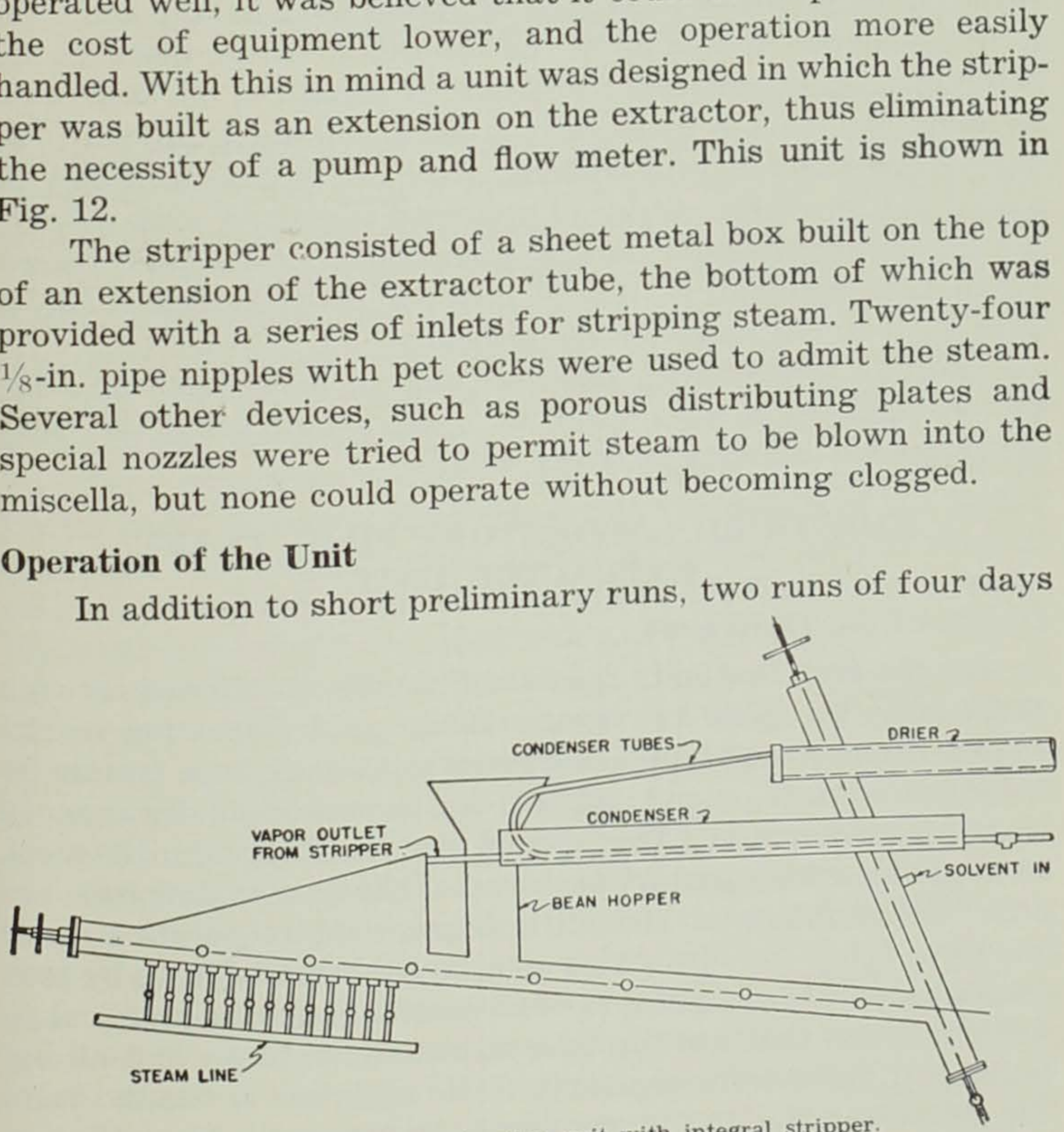

Fig. 12. Modified extraction unit with integral stripper.

each were made. In the first, meal with an oil content as low as 0.6 per cent was produced. None of the oil was completely free of solvent and considerable foaming occurred.

It was decided to apply more heat to the stripper in an effort to remove all of the solvent. Approximately 26 feet of ⅜-inch copper tubing was coiled around the outside of the stripper and one length placed inside. The stripper was lagged to reduce heat losses and steam at 90 pounds per square inch pressure was applied to the tubing for heating.

In the second run, operating at about 13 lb. of beans per hour, part of the meal was extracted to 0.6 per cent oil content although some of it ran as high as 4.2 per cent. Some of this variation may have been caused by rather poor flaking and some to irregularities in feeding of the solvent. Accurate tests for the determination of very small amounts of solvent in oil, used later, were not available in these early studies, making it difficult to be sure of the exact solvent content of the oil. In general it may be stated that probably all the oil produced contained some solvent, but that produced under the best conditions contained only traces. When the temperature in the stripper dropped below 120°C. (248°F.) foaming caused some difficulty.

Results secured with this unit were in many respects satisfactory and led to the design of a third unit using the same principles but differing in many details.

THE THIRD LABORATORY PILOT PLANT EXTRACTOR UNIT

Design of the Third Unit

In the first two units the flaked beans were carried through the solvent by screw conveyors. While good extraction results were secured, it was found that screw conveyors have certain inherent disadvantages for this purpose. It was originally assumed that the solvent would flow around the spiral through the meal, thus giving a long extraction travel. This type of flow does not occur for two reasons: first, it is impractical to operate a screw conveyor completely full, thus a space for the solvent to by-pass the flakes is always present; and second, it is not practical to make a screw that fits the tube so closely as to prevent all by-passing of the solvent around it. Some agitation is secured from the screw but, due to the low speed, this is small. The agitation

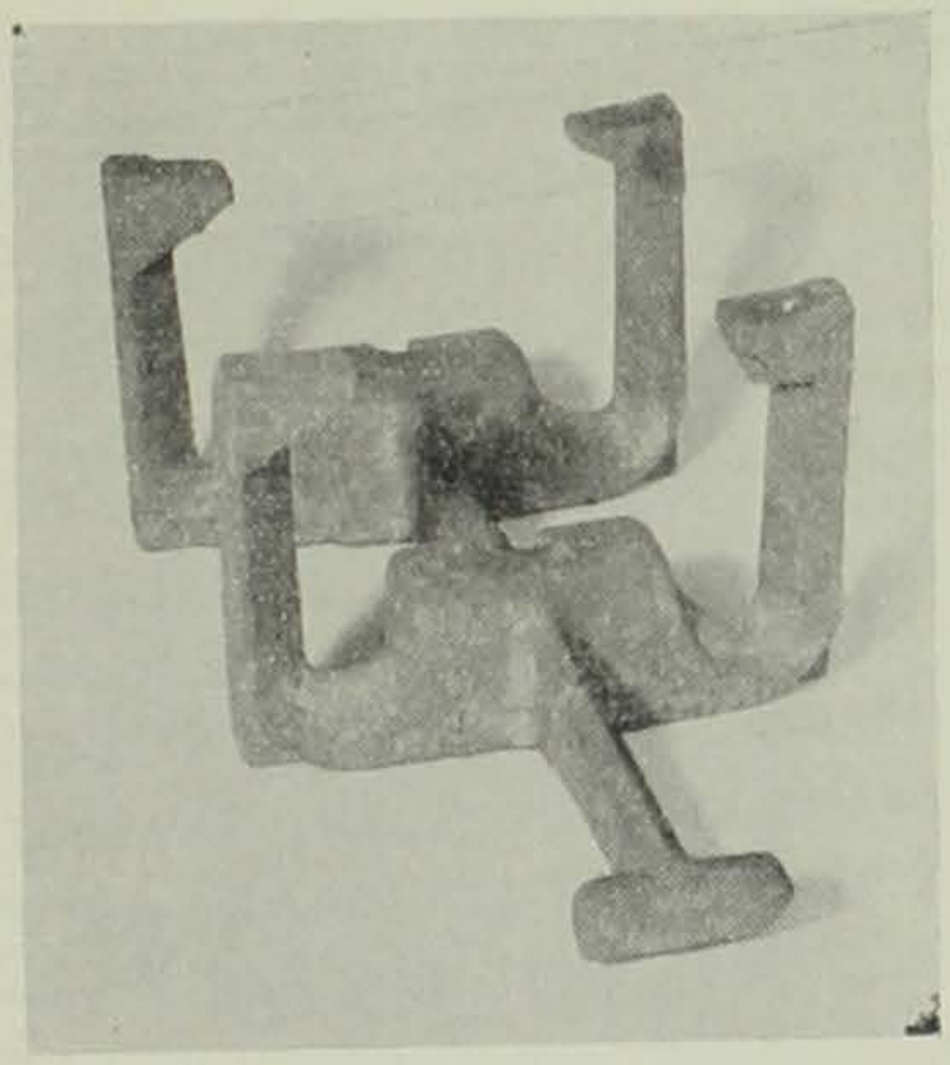

Fig. 13. Detail of Redler chain links.

causes some breaking up of the flakes and this is undesirable in the extraction zone since it increases the amount of total fines, thus producing more fines in the miscella leaving the extractor.

The ideal extraction conditions should be present when the flakes are moved through the extraction casing without agitation, in a uniformly dense mass completely filling the cross-section of the casing, and when solvent flows through this mass of flakes with uniform streamlined flow unrestricted by the conveying device. Such conditions are more nearly approached by the use of Redler chain than by a screw conveyor. The details of the Redler chain are best understood by reference to Fig. 13. The original extractor using this chain is shown in Fig. 14. The casing in which the continuous chain travelled was a slightly-tilted rectangular loop mounted with the feed end high. The inside dimensions of this casing in the extractor portion were 4 in. by 5 in. The upper horizontal casing was enlarged to a cross section 6½ in. by 6½ in. and was provided with a circular steam jacket designed to operate at 60 pounds steam pressure. The chain travelled counter-clockwise around the loop, driven by a sprocket at the upper turn at the high end. Power for the extractor was supplied by a 5 H.P. electric motor through a speed reducer and a series of sprockets and roller chains. Both the motor and speed reducer were much oversize but were used because actual power requirements were not known at the beginning of the work.

The extractor was suspended on cross beams between two

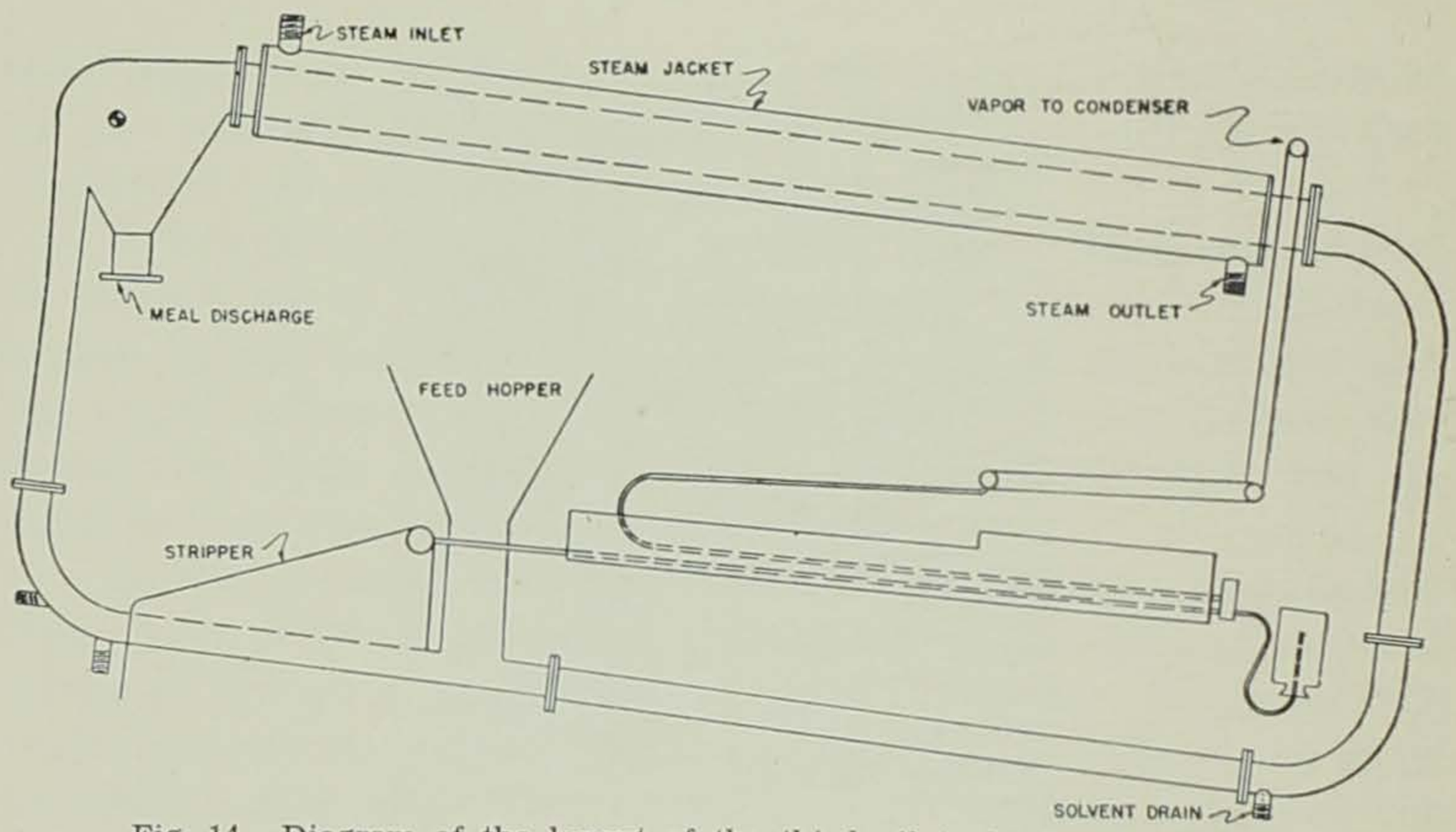

Fig. 14. Diagram of the layout of the third pilot plant extraction unit.

parallel horizontal steel I-beams. A floor was built on top of these beams on either side of the extractor. Another floor was suspended by rods from the beams about 7 feet below the upper floor, thus giving access to all parts of the equipment.

On the bottom of the extractor casing in the stripper section, a $\frac{3}{8}$ inch thick steel plate was welded to provide sufficient thickness for attaching the steam valves used for admitting the stripping steam. These valves were constructed from brass rod and standard $\frac{1}{4}$-inch needle valves. The valve bodies were replaced by special bodies turned from the rod and so constructed that the outlet opening was at right angles to the inlet and opposite the controlling hand wheel. The valves were screwed into the bottom of the extractor so that the end of the valve was flush with the inside face of the plate, and the end of the needle of the valve just reached through the outlet opening. Any dirt or sediment in the valve was thus forced out when it was closed.

The stripper section was heated by two units, each consisting of 24 vertical pieces of $\frac{5}{8}$-inch copper tubing manifolded at top and bottom into copper tubing connected through a steam trap to the steam supply. The outside was lagged with magnesia block insulation. Two outlets for the soybean oil were provided, one at the bottom, just beyond the stripper section, and one in the bend.

The vapors from the stripper passed out through two pipes near the top. Two $\frac{5}{8}$-inch copper tubes led from the two pipes to

the condenser which consisted of seven $\frac{5}{8}$-inch copper tubes submerged in a horizontal sheet metal box 6 inches by 7 feet. The bottom four of these tubes were connected to the stripper. The upper three tubes were a continuation of horizontal tubes carrying vapors from the drier to horizontal runs submerged in the cooling water.

The drier was the upper horizontal jacketed section of the Redler chain casing which was enlarged to $6\frac{1}{2}$-inches by $6\frac{1}{2}$-inches cross section to allow space for the vapor to escape to the outlet at the lower end. The barrel valve used on previous units was installed on the drier discharge together with sufficient sheet metal pipe to reach a bag on the floor below.

The operation of the apparatus was as follows: the flaked beans were fed into the hopper and dropped down into the miscella-filled extraction casing through which they were carried to the vertical section. Here they emerged from the solvent, draining as they passed upward to the drier section. In the drier the solvent was volatilized and the meal dropped out through the barrel valve. The miscella passed into the stripper where it was heated by the steam coils on the side and subjected to the steam distillation or stripping action of steam blown in through the valves in the bottom. The stripped oil passed out the discharge pipe at the end. The solvent vapor, together with water vapor from the injected steam, passed upward in the stripper to the outlet pipes and flowed out into the condenser. Solvent vapor and water vapor from the drier also flowed down to the condenser and were condensed in a separate set of tubes. The condensate (water and trichloroethylene) passed into a common manifold and into the separator where the water and solvent separated by gravity, the former going to the sewer, the latter directly back into the extractor unit. Fresh solvent was added from a glass separatory funnel directly into the pipe entering the extractor to compensate for any losses from the system.

Extraction of the Oil

Two major problems were involved in the extraction: feeding the flaked beans into the extractor and removing the oil or miscella. Poor operation of parts of the process frequently caused abnormal extraction results. Because of inadequate drier capacity during many of the earlier runs it was necessary to operate the extractor intermittently, thus interfering with true continuous

counter-current flow. The residual oil left in the meal under these conditions varied from 0.4 to 11.0 per cent.

The flaked soybeans originally were fed by gravity from a sheet-metal hopper into the extractor. Difficulty was encountered in filling the extractor casing for two reasons: first, flaked soybeans do not sink readily in trichloroethylene-soybean oil mixtures partly because of the high specific gravity and viscosity of the liquid, and partly because of the slowness of "wetting" (that is, the removal of the air films between flakes and substitution with miscella); and second, soybean flakes, especially if moist on the surface, do not flow downward readily through straight-sided or hopper conduits, but tend to arch and stick. Increasing the height of a column of flakes in a vertical tube or casing is of no value in producing a suitable head since the friction of the flakes on the sides offsets any advantage of weight due to height. Consideration of these points lead to the use of agitation. Simple punching with a stick showed some advantage although care had to be exercised to secure uniform flow and to avoid undue packing. An agitator was constructed using "puncher rods" consisting of ½-inch steel rods to which were welded short lengths of flat steel strip at an angle of about 30°. Ten of these pieces were welded to each of two 3-foot rods, the bottom end of each piece extending outward from the rod. These were given a vertical reciprocating motion by a suitable power-driven crank shaft arrangement, the number of punching movements varying from 1 to 6 per minute.

Before using this feeding device about 20 pounds of meal were fed per hour to the extractor when the Redler chain was operated at 2.5 inches per minute. With the feeding device, up to 60 pounds per hour were fed at 3 inches per minute and 70 pounds at 4.6 inches per minute. At the speed of 4.6 inches per minute, 50 pounds of beans per hour, and a miscella of 9.8 to 23.0 per cent oil, the residual oil in the meal ranged from 0.5 to 1.2 per cent. When 70 pounds per hour were processed at the above chain speed and a miscella of 20.5 to 24.4 per cent oil, the residual oil in the meal was 1.5 to 2.0 per cent. The miscella data given are not directly comparable with later data since some concentration of the miscella occurred in the stripper section before leaving the extractor, whereas later data refers to unconcentrated miscella.

After the addition of an outside stripper to replace the in-

tegral stripper section, it seemed desirable to feed in the flakes at a point farther back in the extractor and thus secure greater extraction length. A sheet metal feed spout was attached above the sprocket and the flakes were fed into this with a hand scoop. Flakes were dumped into the hopper during a run in 25 lb. increments, always keeping the level no lower than the bottom of the hopper to insure an effective seal against solvent vapor loss. Certain types of flakes arched over in the hopper, but in general did not cause much difficulty in feeding.

The speed of the Redler chain was increased eventually to 11.5 inches per minute. At this speed as much as 160 lb. per hour of flakes were extracted. At 140 lb. per hour and a miscella containing 20 per cent or less oil, the residual oil in the meal was less than 1 per cent, usually about 0.7 per cent. Under the above conditions about 20 gallons of solvent were circulated per hour.

The solvent inlet was about 1 foot above the miscella outlet in this design using an external stripper, thus allowing the solvent to flow through the meal by gravity. Since the Redler chain and the flakes moved counter current to the solvent flow, the chain tended to retard the flow, and thus raised the level above that necessary to cause a flow of solvent through a stationary mass of flakes.

Removal of Solvent from the Oil

Attempts were initially made to remove the solvent from the oil in the integral stripper in the same manner as in the second model. It was found impossible to remove all the solvent from the oil in this way, and the tubular stripper and condenser used with the first large-scale unit were connected to take the miscella from the integral stripper. The copper tube condenser connected to the integral stripper was removed so that all of the solvent vapor ran to the new condenser and the condensate from this condenser ran to the solvent-water separator previously described. In an effort to prevent fines from being carried out with the miscella and fouling the stripper, a 60-mesh screen was placed in the old stripper section. This failed to retain sufficient fines to prevent fouling, while a finer screen soon clogged and could not be used.

The feed was then changed from the original hopper to a new feed inlet at the top of the vertical section of the extractor

(see details under extraction studies). The stripper chamber above the chain was blocked off and the old hopper closed. Two miscella take-off boxes were now installed, one on each side of the vertical casing. The open sides of the boxes next to the chain were covered by a 40-mesh screen, with the surface of the screen flush with the inside of the casing. These miscella outlets functioned satisfactorily, the miscella flowing out through the screen in a steady stream usually rising no higher than one inch on the screen. The openings in the screen were kept open by the wiping movement of the chain.

In the operation of the stripper steam was blown into the bottom of the column and both solvent and water vapor were taken off from the vertical center of the stripper. Under good operating conditions no solvent could be tasted in the oil from the stripper. It was assumed, therefore, until the pyridine chemical test was used (see Appendix A), that the stripped oil was solvent-free. Upon checking by this new method it was found that some of the oil produced contained as much as 2 per cent solvent. By increasing the steam pressure around the tubes from 50 to 90 p.s.i. and blowing in large amounts of stripping steam, it was possible to strip the usual miscella containing 20 per cent oil completely. This was not considered practical for commercial operation because the steam consumption was high, the oil was dark colored, and the allowable variation in rate of operation was small.

Packed columns were next investigated as suitable equipment for the final stripping of the solvent from the oil. Since excellent counter-current contact may be secured between a descending liquid and an ascending gas or vapor in packed columns, they are sometimes used in steam distillation and stripping operations. Various types of filling material are used in these columns with Raschig rings (hollow ceramic cylinders having their length equal to their diameter) being considered excellent. Preliminary experiments on packed-column stripping were carried out in a column built from sewer tile and packed with Raschig rings. While this column did not prove practical because of difficulties with vapor leaks it gave such promising results that a steel column was constructed.

The new column (Fig. 15) consisted of a 9-foot length of 8-inch steel pipe flanged at both ends and set vertically. Four 1-inch pipes closed at the upper end extended upward from the bottom

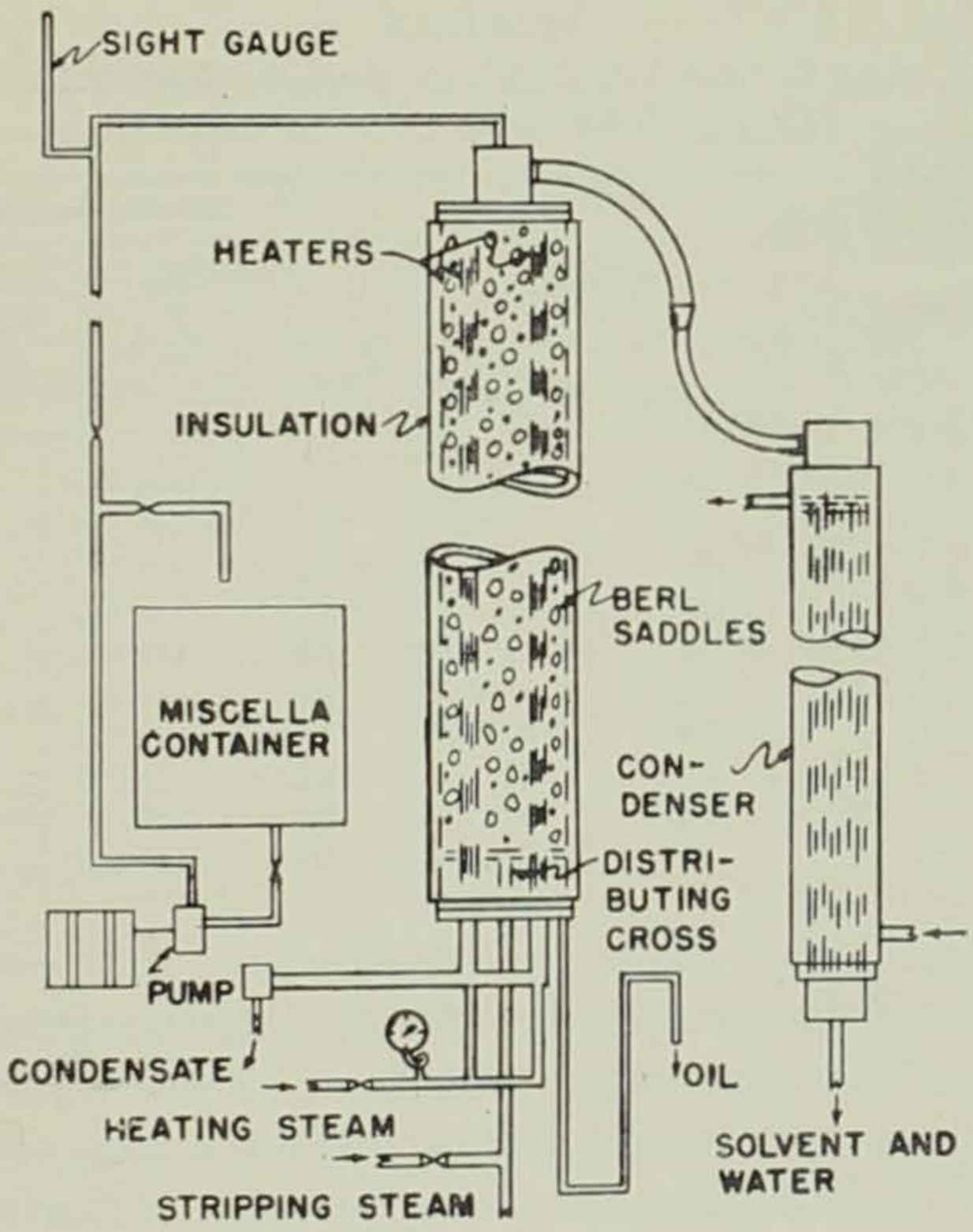

Fig. 15. Packed-column stripper using Berl saddles.

blind flange to within about 6 inches of the top. Inside each of these pipes and extending nearly to the top was a ¼-inch pipe. Steam entered the ¼-inch pipes and condensate was removed from the bottom of the 1-inch pipes through suitable connections and a trap. Since the heating pipes were all attached to the bottom flange, the whole assembly could be dropped down by removing the flange bolts. Stripping steam was blown in through a pipe entering at the center of the bottom flange and connected to a distributing cross. A pipe tee was welded to the top flange with the run set vertically and a vapor line ran from the side of the tee to the condenser. The miscella was pumped from a 30-gallon tank on the floor through a ¼-inch pipe which entered the stripper through the top of the tee. The stripper was originally filled to a height of about 5 feet with 1-inch Raschig rings. Above these were ¾-inch rings to within 6 inches of the top.

A series of runs were made using various concentrations of trichloroethylene in soybean oil from which representative data are given in Table 11. The results of these runs indicated that

TABLE 11

RESULTS OF STRIPPING IN PACKED COLUMN.
PACKING MATERIAL: RASCHIG RINGS

No. Run	Solvent in input %	Length of run hours	Rate of input lb. per hr.		Steam Requirements			
					Heating		Stripping amount lb. per hr.	Total per lb. of oil
			Miscella	Oil	Pressure p.s.i.	Amount lb. per hr.		
1	2.0	3	54.7	54.	40	10.7	20.0	0.6
2	2.0	3	53.3	52.2	40	10.7	17.2	0.5
3	4.0	3	59.	57.	50	10.7	30.8	0.7
4	4.0	3	47.	45.	40	10.7	29.7	0.9
5	10.0	3¾	58.	52.	50	10.7	31.9	0.8
6	20.0	3½	57.	46.	50	10.7	28.2	0.8
7	30.0	2	54.5	38.	50	10.7	38.7	1.3
8	20.0	2½	52.0	42.	50	10.7	22.0	0.8
9	20.0	2	46.3	37.	45	10.7	19.8	0.8
10	14.0	4	59.2	51.	50	10.7	18.8	0.6

the solvent could be stripped from miscella containing 20 per cent trichloroethylene at the rate of 40 lb. of oil per hour.

The packed-column stripper was then arranged to take miscella from the old falling-film stripper. No steam was injected into the bottom of the latter, and in most runs the steam on the bottom tube section was cut off. The miscella, concentrated to approximately 80 per cent oil, flowed over two baffles in the pan at the bottom of the stripper into the miscella tank on the floor. From there it was pumped to the packed-column stripper. As would be expected from the data in Table 11, the capacity of the packed column was larger than necessary for stripping the miscella from the pilot-plant extractor. After operating the packed column as a part of the extraction system for a series of runs in which a total of 6250 lb. of beans were processed, it was opened and the rings examined. They were found to be badly fouled with fines.

The column was then filled with 1-inch Berl saddles (U-shaped ceramics). There were two reasons for this change. It was obvious that Raschig rings could not be satisfactorily used with unfiltered miscella from the extractor and it was hoped that the fines in the miscella would wash through the Berl saddle bed. The second reason was the higher efficiency and capacity claimed for the saddles. Actual operation of the column with the saddles in the pilot-plant showed that it was not practical to operate with-

TABLE 12

RESULTS OF STRIPPING IN IMPROVED PACKED COLUMN WITH BERL SADDLES

Run No.	Solvent in input %	Length of run hours	Rate of input lb. per hr.		Steam Requirements	
					Heating	
			Miscella	Oil	Pressure p.s.i.	Stripping amount lb. per hr.
1	5	3 1/2	126.4	120	40	66.5
2	10	3 1/6	133.4	120	40	63.
3	15	3 1/6	130.7	111	35	63.5
4	20	3	145	116	40	63.5
5	20	2	150	120	55	62
6	20	4	125	100	35	66
7	30	2 1/2	138.6	97	40	63.5
*8	18	1 1/2	146.4	120	40	62

* Run with complete plant in operation.

out first filtering the miscella. A horizontal plate filter (Sparkler) having a 1 square-foot capacity operating with a small rotary pump at pressures up to 50 p.s.i. was installed in the line between the extractor and the falling-film evaporator. Paper was used as a filtering medium. The filter operated well, filling almost completely with fines before reaching the maximum operating pressure of 50 p.s.i. In general, with the exception of emulsion trouble which will be discussed later, this arrangement operated very well and produced an oil free from solvent. Operating data on the column using Berl saddles is given in Table 12. Higher capacities were secured with the saddles than with the Raschig rings but part of this increase may have been due to the use of filtered miscella with no fines to foul the column and reduce the capacity.

Although the packed column proved to be excellent for final stripping of the oil, for efficient operation it needed a miscella containing more oil than that coming directly from the extractor. A falling-film evaporator or tubular stripper, operated without injection of steam directly into the miscella, may be used to concentrate the miscella from the usual 20 per cent oil from the extractor to 80 per cent or higher as input for the packed-column stripper, but has certain disadvantages. As has been noted previously, the miscella contained small amounts of fine meal which eventually fouled the tubes, reducing the efficiency considerably. Even filtered oil would tend to bake on the hot tubes, particularly

when the amount of miscella flowing was small, as at the beginning or end of a run. It was difficult to secure a uniform distribution of the miscella over all the tubes thus leading to the fouling of certain tubes, particularly when the miscella flow to the evaporator was not uniform.

In an effort to develop concentrating equipment free from these disadvantages, a study was made of the application of the climbing-film evaporator principle to the concentration of miscella. The climbing-film principle is used industrially in the Kestner evaporator. This is a vertical-tube evaporator in which the liquid to be evaporated passes upward by climbing-film action through steam-heated tubes. The tubes are from 1¼ to 2½ inches in diameter and 10 to 20 feet long with the liquid level 2 to 3 feet above the bottom of the tubes.

After some preliminary experimentation, the laboratory apparatus shown in Fig. 16 was set up. A glass tube about 7 ft. high and 9 mm. internal diameter was wound with resistance wire as a heater and mounted vertically. The feed was placed in a 1 liter graduated cylinder connected by a stopper and glass tubing to a dropping funnel which in turn was connected to the preheater. The tube connecting the cylinder and the dropping funnel

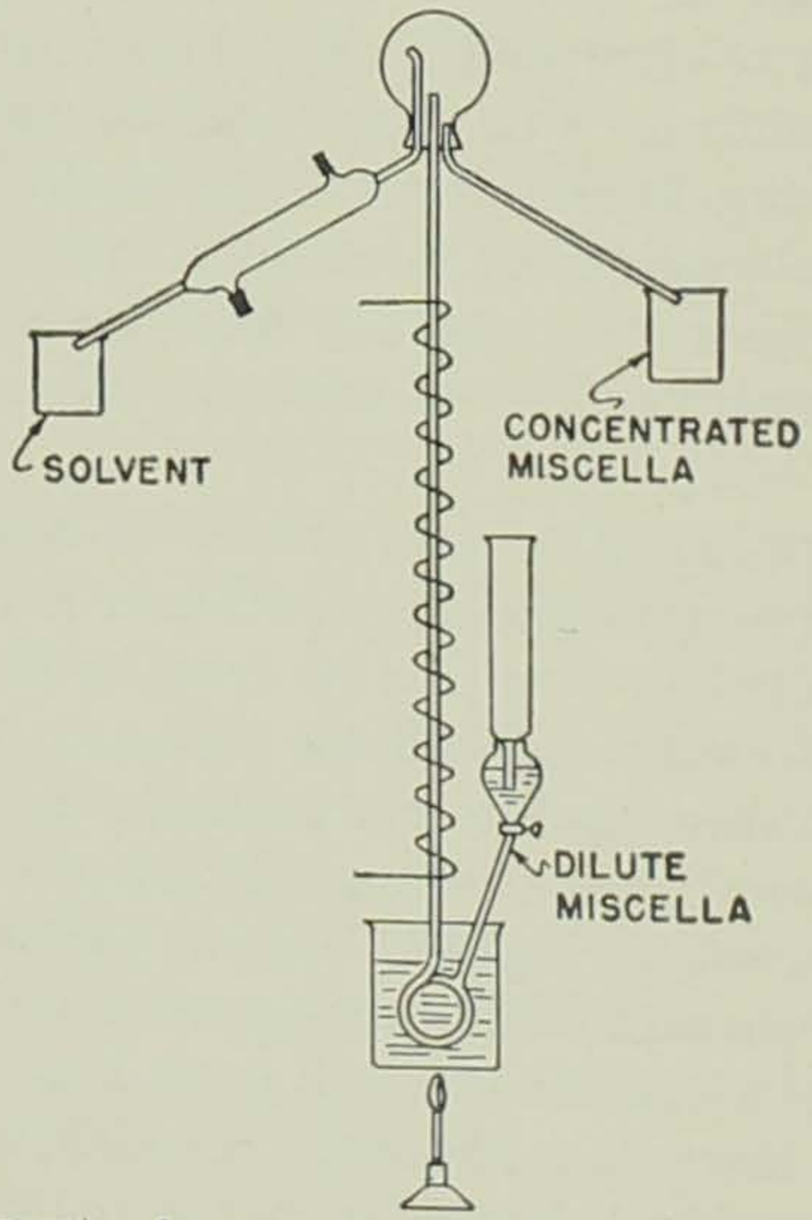

Fig. 16. Laboratory apparatus for testing the principle of the climbing-film evaporator.

extended below the level of the liquid in the dropping funnel, giving a constant liquid heat. The preheater consisted of a loop of copper tubing immersed in a beaker of water which was kept slightly below 100°C. (212°F.). An inverted 3-necked flask was attached to the top of the tube to act as a flash chamber. The vapor was removed through one neck and condensed in a glass condenser. The concentrated miscella flowed out the other neck through a trap of glass tubing.

In operation the liquid was observed to boil at the surface in the long tube. As it boiled disks of miscella formed with vapor between, the disks rising with increasing velocity towards the top of the tube, due to the increased vaporization of solvent from the two surfaces of the disks as well as the wall of the tube. The liquid left the tube at a high velocity and impinged on the bottom of the flask. Results of operation of this unit using both 20 and 30 per cent miscella are given in Table 13. Since these results showed promise, the work was continued on a larger scale in metal equipment.

The first evaporator consisted of a ½-inch vertical pipe with a steam jacket consisting of 12 feet of 1½-inch pipe. The top of

TABLE 13

THE CONCENTRATION OF MISCELLA IN A CLIMBING-FILM EVAPORATOR

Input percent oil	Product cc per min.	Percent oil	Equivalent oil rate lb. per hr. for 1 in. tube
20	2.3	71.0	1.7
20	5.1	74.0	4.0
20	13.0	59.7	8.8
20	17.0	56.0	11.0
20	16.4	65.9	12.0
20	28.0	39.7	13.8
20	27.0	49.7	16.0
20	31.0	44.4	16.7
20	34.8	39.4	17.1
20	43.1	35.0	19.2
20	56.3	32.7	23.7
30	8.1	80.2	6.8
30	10.6	81.6	9.0
30	12.2	86.9	10.8
30	18.0	71.5	14.1
30	26.8	63.9	19.2
30	32.0	62.9	20.2

the small pipe extended into a can which served as a flash chamber. The concentrated miscella passed out of the flash chamber through a V-bend to a collecting can and the vapor was carried to a condenser. The results of the first trials on this equipment using miscella containing 20 per cent oil are shown in series 1, Table 14. In these first runs there was considerable surging of the miscella and considerable vapor collected in the feed line. A larger flash chamber was installed and the feed was run in directly instead of siphoning it in. The runs of series 2 and 3 were made next, run 3 with a 20 instead of a 12-foot jacketed pipe. Before series 4 the feed tank was replaced with a pipe with an overflow so as to insure a constant head. Series 5 shows the effect of preheating the input to 60°C. (140°F.). Because of the short time of the runs, it was difficult to iron out the irregularities incident to starting and stopping and secure results as consistent as might be desired. In general, the data indicate that increasing the head

TABLE 14

THE CONCENTRATION OF MISCELLA IN A CLIMBING-FILM EVAPORATOR — ½-INCH TUBE*

Series No.	Length of pipe in feet	Steam p.s.i.	Head in inches	Time in minutes	Oil in product per cent	Lbs. of oil per hr. (20% solution)
1a	12	80	12.5	105	89	13.0
b	12	80	14.5	90	88	14.1
c	12	75	17.5	45	87	13.9
d	12	65	21.0	67	84	19.2
2a	12	77	25.5	85	75	21.0
b	12	77	16	90	83	15.5
3a	20	77	25	105	81	18.0
b	20	77	31	135	83	14.8
c	20	74	31	115	84	17.0
d	20	74	36	100	85	18.9
e	20	74	48	90	75	20.0
4a	20	76	36	45	81	27.0
b	20	78	36	85	87	20.6
c	20	78	36	110	87	18.0
d	20	76	42	90	80	20.1
e	20	74	42	80	81	20.4
5a	20	76	42	60	84	22.6
b	20	76	42	85	89	21.7
c	20	78	36	85	92	20.7
d	20	—	36	100	89	16.5
e	20	78	42	75	87	21.9

* ½-inch standard iron pipe, 0.62 in. diam.

(that is, the height the miscella stands in the bottom of the tube when not operating) increases the amount processed but decreases the percentage of oil in the product. Increasing the length of the heated portion from 12 to 20 feet had little effect. Preheating the feed to 60°C. (140°F.) increased slightly the oil content of the product. It may be concluded that a 20-foot ½-inch pipe climbing-film evaporator will concentrate 20 lb. per hour of 20 per cent miscella to 85 per cent oil-content.

Data from a 1-inch pipe jacketed with 11 ft. 3 in. of 3-inch pipe are shown in Table 15. The results from series 1 show the same tendency of greater head to increase the amount and decrease concentration of finished product observed with the ½-inch pipe. Inadequate condenser capacity, which was remedied in series 2, may have slightly reduced the capacity of the equipment in series 1. This evaporator concentrated miscella equivalent to 45 lb. of oil per hour from 20 to 80 per cent oil.

Two evaporators each having 20 feet of jacketed ½-inch pipe and discharging at the top into a flash chamber were connected to the pilot-plant extractor to concentrate the miscella, prior to reaching the packed column. The evaporators operated with a regular pulsating movement of the miscella into the flash chamber and a corresponding movement back into the miscella line. The pulsating movement was transmitted through the air from the miscella feed tank to the miscella outlet screen resulting in

TABLE 15

THE CONCENTRATION OF MISCELLA IN A CLIMBING-FILM EVAPORATOR — 1 IN. TUBE

Series No.	Steam p.s.i.	Head inches	Oil in feed per cent	Oil in product per cent	Temperature of input °C	Lb. of oil per hr.
1.	74	16	20	82	Room	30.0
	67	22	20	84	Room	34.5
	72	22	21	82	Room	41.6
	78	28	20	79	Room	47.4
	...	28	20	71	Room	51.6
	80	34	21	72	Room	57.9
2.	74	22	22	84	Room	46.0
	75	22	20	80	Room	45.4
	83	22	22	83	Room	45.6
	74	14	22	82	60 (140°F.)	49.0
	74	22	22	75	60 (140°F.)	71.5
	80	22	20	76	60 (140°F.)	56.5

a pulsating flow through the screen. This not only helped to keep the screens open for the miscella flow, but resulted in less fine meal coming through in the miscella. For example, when using the falling-film stripper, an average of 5 runs showed 0.78 per cent of the weight of beans processed as dry oil-free fines in the miscella while 2 runs using the climbing-film evaporator produced an average of 0.24 per cent dry oil-free fines.

When the climbing-film evaporators were installed as a part of the pilot plant, one of the bag filters used in the first pilot plant was connected between the flash chamber and the packed-column stripper, filtering the miscella by gravity flow only. Good results were secured from this bag filter, but both Sweetland and Kelley leaf filters were tried since these are standard filters and easier to empty. The Sweetland filter had nine 7-inch leaves and, after operating in a run in which 1792 lb. of beans were processed, contained 6½ lb. of dry oil-saturated fines which filled it about one-third full. The Kelley filter was 20 inches long by 9 inches in diameter with 3 leaves having an area (one side only) of 324 square inches. On a run of 2532 lb., it contained 11 lb. of dry oil-saturated fines, but was not completely filled. When the miscella is filtered between the evaporator and the stripper, a smaller volume of liquid is filtered than when filtered ahead of the evaporator. The miscella at this point is hot so that filtration is rapid.

Emulsion Difficulties

When the packed-column stripper was first set up in connection with the extractor it was connected to a separate condenser, the condensate from which ran into a pail. After the pail became full, the water was allowed to overflow until the trichloroethylene level became high enough to necessitate emptying into another container, and it was noted that a white emulsion formed in the water layer. It was observed that this emulsion was usually considerably heavier and more stable when operating the stripper in conjunction with the extraction apparatus than when stripping a miscella produced by mixing oil and trichloroethylene together outside the extractor. Since the stripper had capacity considerably in excess of that necessary to handle the miscella from the pilot plant, the stripping studies carried out on the "synthetic" miscella were ordinarily at a rate considerably higher than on the plant miscella. It was at first thought that variation in emulsion

might be associated with the variation in input. However, it was found that freshly extracted oil mixed with solvent and stripped the first time produced more emulsion than that which had been stripped several times following successive readmixtures with solvent.

Examination of the emulsion indicated that it was composed of soybean oil, water, and trichloroethylene. The trichloroethylene was dissolved in the oil and in droplets held mechanically in the emulsion. The amount of oil and trichloroethylene dissolved in the oil was probably not sufficient to be of much economic importance in commercial operation, however, the amount of solvent carried out in the form of droplets in the heavy emulsion was sufficient to be a serious loss. The heavy emulsion also clogged the tubes of the condenser sufficiently to prevent proper operation.

Since the emulsion was essentially oil and water, it was assumed that the oil, since it is not volatile at the temperatures used, must have been carried over mechanically by the steam. It hardly seemed probable that the vapor velocity in the column was sufficient to entrain the oil directly. If, however, the oil were spread into a very thin film as would be the case if foaming occurred, it might be carried out in the form of colloidal droplets at a low vapor velocity. Ineffective efforts to throw out the oil by means of sheet metal baffles and a glass wool filter indicated that the entrained oil droplets must have had very small dimensions. Since the oil contains phosphatides which are excellent emulsifying agents, it would be expected that the oil in a highly dispersed condition such as a fine colloidal mist would readily form an emulsion with water, particularly if the latter is in the form of a condensing film with a high ratio of surface to volume.

To secure additional information in regard to the emulsion formation, a glass stripping column consisting of a glass tube 2 in. in diameter by 48 in. high packed with ½-inch Raschig rings and heated electrically by resistance wire was set up and a glass Liebig condenser used to condense the vapor. Several interesting facts were observed. When steam was blown through the hot column which had previously contained miscella, but was now empty except for such oil as might be retained on the surface of the rings, a heavy type of emulsion similar to that clogging the condenser in the larger unit was observed. Apparently some material

causing the emulsion was being steam distilled out of the oil present on the surface of the rings.

After the rings had been cleaned, expeller-produced oil with low lecithin content and free from trichloroethylene was run through the column to coat the rings. When steam was blown in after draining the oil from the column, practically no emulsion was formed. When commercial lecithin was added to the oil and the procedure repeated, a large amount of emulsion was formed. Apparently the emulsion was caused by some product from the lecithin or associated phosphatides which was steam distilled into the condenser. Since lecithin decomposes at 100°C. (212°F.) and the emulsion begins to form at column temperatures of 130°C. (266°F.) or higher, it seemed probable that the emulsifying agent was a decomposition product of lecithin or other phosphatides.

When miscella was run through the stripper in the usual manner the emulsion was not deposited in the condenser. When pure oil was run into the column with steam, even when heated to scorching temperatures, no emulsion was deposited in the condenser. Apparently, part of the fouling of the condenser was caused by this material being steam distilled from the residual oil remaining on the column packing from the preceding run. However, it seemed evident that most of the emulsion which collected in the solvent-water separator was caused by entrained oil which contained sufficient phosphatides to produce an emulsion.

It was found by experiment that the emulsion could be broken and the trichloroethylene recovered by boiling. Since the heat content of the vapors was sufficient to revolatilize any part of the condensate, experimental work was undertaken in an effort to break the emulsion and revolatilize out the solvent without the addition of outside heat.

In the first laboratory apparatus for this purpose a three-hole round-bottom flask was mounted on its side. A vertical glass Liebeg condenser was connected by glass and rubber tubing to the lower hole. Tubing from the middle hole ran to the drain and a horizontal condenser was connected to the upper hole. Vapor from the stripper passed down into the vertical condenser where it was partially condensed, the mixture of liquid and vapor going to the bottom hole of the flask. When the amount of cooling water

on the condenser was properly adjusted, trichloroethylene and water vapor passed out the upper hole into the second condenser where they were condensed. Water containing the oil from the emulsion but free of solvent passed out the middle hole. This apparatus was used with success on a small stripper operating on a mixture of oil and solvent. Larger apparatus constructed from a steel tank and metal condensers was designed to give the same results when used with the regular packed-column stripper operated with the pilot plant. While this apparatus reduced the amount of emulsion, it did not eliminate it as the glass apparatus did.

Apparently, the poorer results secured with the larger apparatus resulted not from the apparatus itself, but from the fact that the glass apparatus was used on miscella from oil which had been run or been in storage long enough to reduce its phosphatide content below that of the freshly-extracted oil. The apparatus was changed so that the emulsion formed could be run back continuously from the separator, and this arrangement was very successful. The water leaving the overflow contained a small amount of oil but averaged slightly less solvent than that soluble in water at room temperature. A modified form of the apparatus using a reflux condenser (Fig. 17) has the advantage of greater compactness and is also self venting through the open top.

The Driers

As originally designed, it was planned to remove all of the solvent from the meal in the upper horizontal Redler chain casing. At least three advantages were expected, the first of which was lower equipment cost. The chain running through the extractor had to be returned through a horizontal casing above the extractor unit. By making the casing larger to allow for vapor removal, heavier to withstand steam pressure, and fitting it with a steam jacket, a drier was provided. The second advantage was the excellent heat transfer from the heated chain to the miscella in the extractor at the flake feeding point. The third advantage lay in the compactness of the combined drier and extractor unit.

As originally operated, the extractor processed about 20 lb. per hour of beans. The drier failed to remove all of the solvent from the meal at this rate of operation, making it necessary to operate intermittently. To increase the capacity, a drier was in-

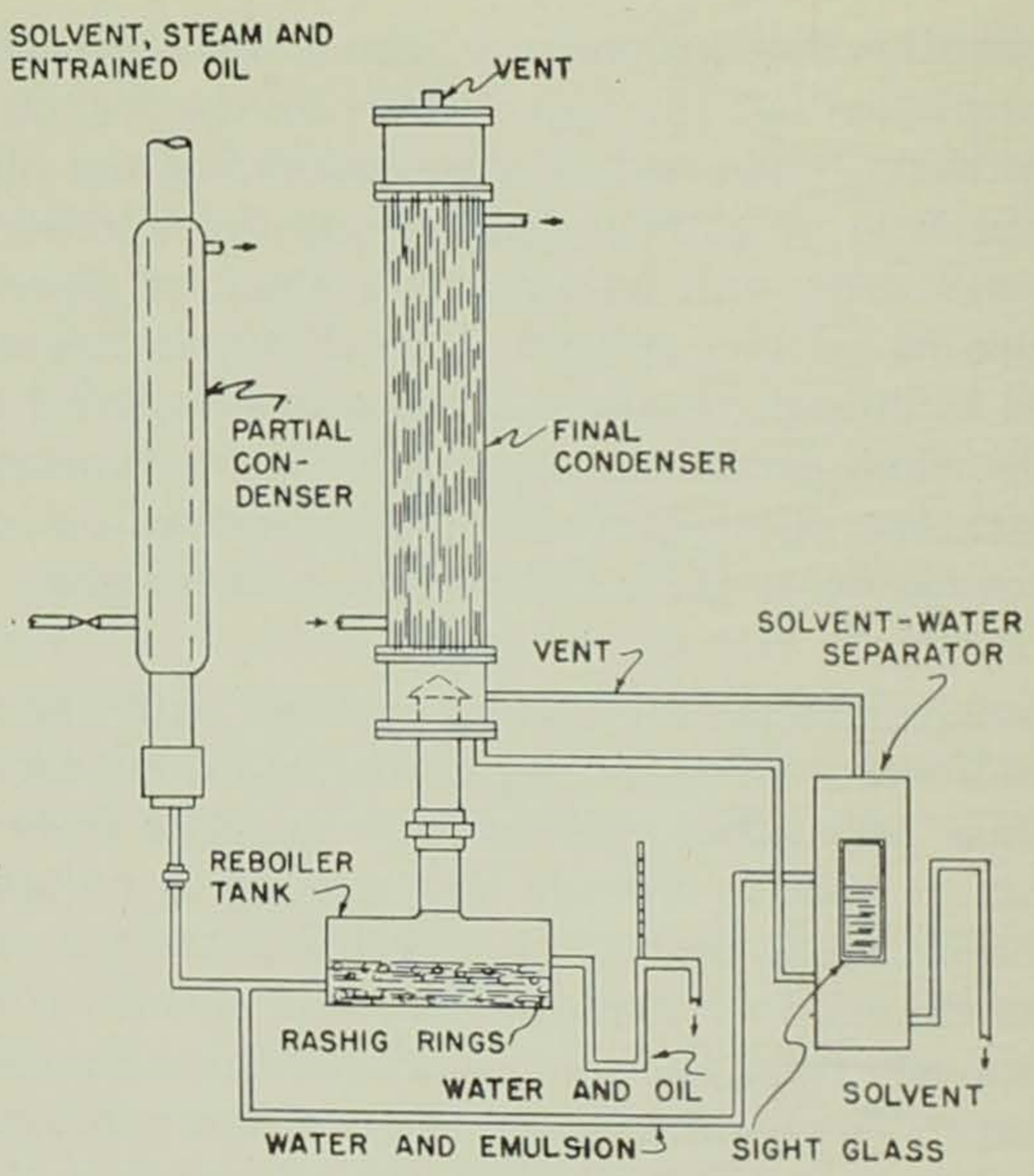

Fig. 17. Improved emulsion-breaking apparatus.

stalled below the original drier to receive the meal from it. This second drier consisted of a jacketed 6-inch pipe in which a modified ribbon screw operated. It was slanted upward towards the outlet end, the vapor being taken off at the low end. The barrel valve outlet for the meal was transferred from the upper drier to the outlet of the lower drier.

The vapors from the upper drier were originally condensed in the copper tube condenser described at the beginning of this section. With the addition of the outside falling-film stripper, a condenser similar to that used on the first pilot plant was installed to condense the stripper vapors. A second condenser (the one previously used in the first pilot plant) was installed to handle the vapors from the two driers. The solvent-water separator previously used with this condenser was installed, and the solvent run to a 10-gallon tank from which it was pumped back into the system by a rotary pump. Vapors from both the driers and the stripper were next run into one condenser only, which proved of sufficient capacity.

The two driers were mounted parallel with nipples and elbow

between. Meal frequently piled up in the elbow completely stopping movement between the driers. A new transition, rectangular in cross section, with its long axis parallel with the driers and connecting the driers with a uniform slant was installed. The meal moved down this very well without clogging.

The clogging in the lower drier was usually caused by the formation of a hard lump of meal on the screw about 4 feet beyond the transition between the driers. The causes of the lump formation were apparently the retardation of flow of the meal, and excessive condensation of moisture on the meal. The screw in the lower drier was made from a ⅜-inch steel rod instead of a flat ribbon. This type of screw produces more mixing action upon the material being conveyed than the flat ribbon, but at a given speed has less conveying capacity. To produce additional mixing action in the drier, short lengths of rod were welded between the arms supporting the ribbon and parallel to the shaft. As was demonstrated in later experimental work, rods of this sort retard the movement of the meal. If the rods do not extend continuously through the drier, the conveyed material is slowed up in movement when it reaches one of the rods, thus piling up at this point. Perfectly dry meal in a dry atmosphere might pile up at points in this way, but would not cause lump formation.

When operating at a rate of 125 lb. per hour, the meal leaving the upper drier still contains about 85 lb. of solvent per hour together with most of the water originally in the beans, usually from 12.5 to 15 pounds. A considerable portion of this solvent flashes into vapor as the meal tumbles down the transition section filling the end of the drier with vapor. More solvent vaporizes as the meal moves forward in the lower drier and as the bulk of the solvent volatilizes out, the temperature rises, and more water vaporizes. The meal at the entrance end and back for some distance, probably at least half way to the discharge end, is at a temperature below the boiling point of water (that is below 100°C., 212°F.) and above that of trichloroethylene (87°C.) (188.6°F.). The vapors pass down out of the lower end of the drier to the condenser. As the water vapor from the warmer part of the drier passes down into the zone which is kept cool by the vaporizing solvent, some of the water vapor wets the meal. The amount of condensed water necessary to form a lump depends upon the amount and physical state of the meal. Meal contain-

TABLE 16
DRIER OPERATION DATA

Steam pressure:
Upper drier — 60 p.s.i.
Lower drier — 90 p.s.i.

Feed to extractor:
a. 125 lb. beans per hour composed of:
90 lb. dry oil-free meal
22.5 lb. oil
12.5 lb. water
b. 219 lb. trichloroethylene

Feed to driers:	Lower Drier	Upper Drier
Dry oil-free meal	90 lb.	90 lb.
Oil in meal	0.9 lb.	0.9 lb.
Water	12.5 lb.	11.8 lb.
Trichloroethylene	132.3 lb.	75 lb.
Trichloroethylene removed	57.3 lb.	75 lb.
Water removed	0.7 lb.	7.0 lb.
Heat transfer coefficient*	2.5	6.3

* B.t.u. per hr. per sq. ft. area per degree Fahrenheit.

ing considerable fine material tends to form lumps more readily than that containing more full-sized flakes. If the vapor velocity is high enough most of the remaining water vapor passes on into the vapor pipe to the condenser either as colloidal water droplets or water vapor.

Several factors may affect the amount of water condensed as: low steam pressure in the drier jackets, air-bound condensers, hot condensers, and clogged vapor lines. Clog-ups in the drier stopped, except for abnormal conditions such as listed above, after the removal of all of the horizontal mixing rods from the conveyor screw.

Although operated at capacities as high as 160 lb. per hour, the drying capacity for complete solvent removal was less than 125 lb. per hour and varied somewhat with the type of flakes. For example, thin flakes dry more readily than thick flakes; dusty flakes less readily than those free of fines; and flakes with moderate amounts of moisture (10 to 12 per cent) more readily than very dry or very wet flakes. Data on drier behavior is given in Table 16.

During normal operation of the apparatus some air would get into the driers and be carried into the condensers. Some of this was carried into the lower drier by the empty pockets of the barrel valve. Unless vented to remove this air, the condenser became air bound and failed to condense the vapors properly. If vented into open air, the non-condensable gases always carried out some solvent vapor. Venting into an absorption tower using soybean oil as an absorbing medium for the solvent vapors was used with the first large scale unit. This method had obvious disadvantages.

The absorption of trichloroethylene vapors by soybean flakes

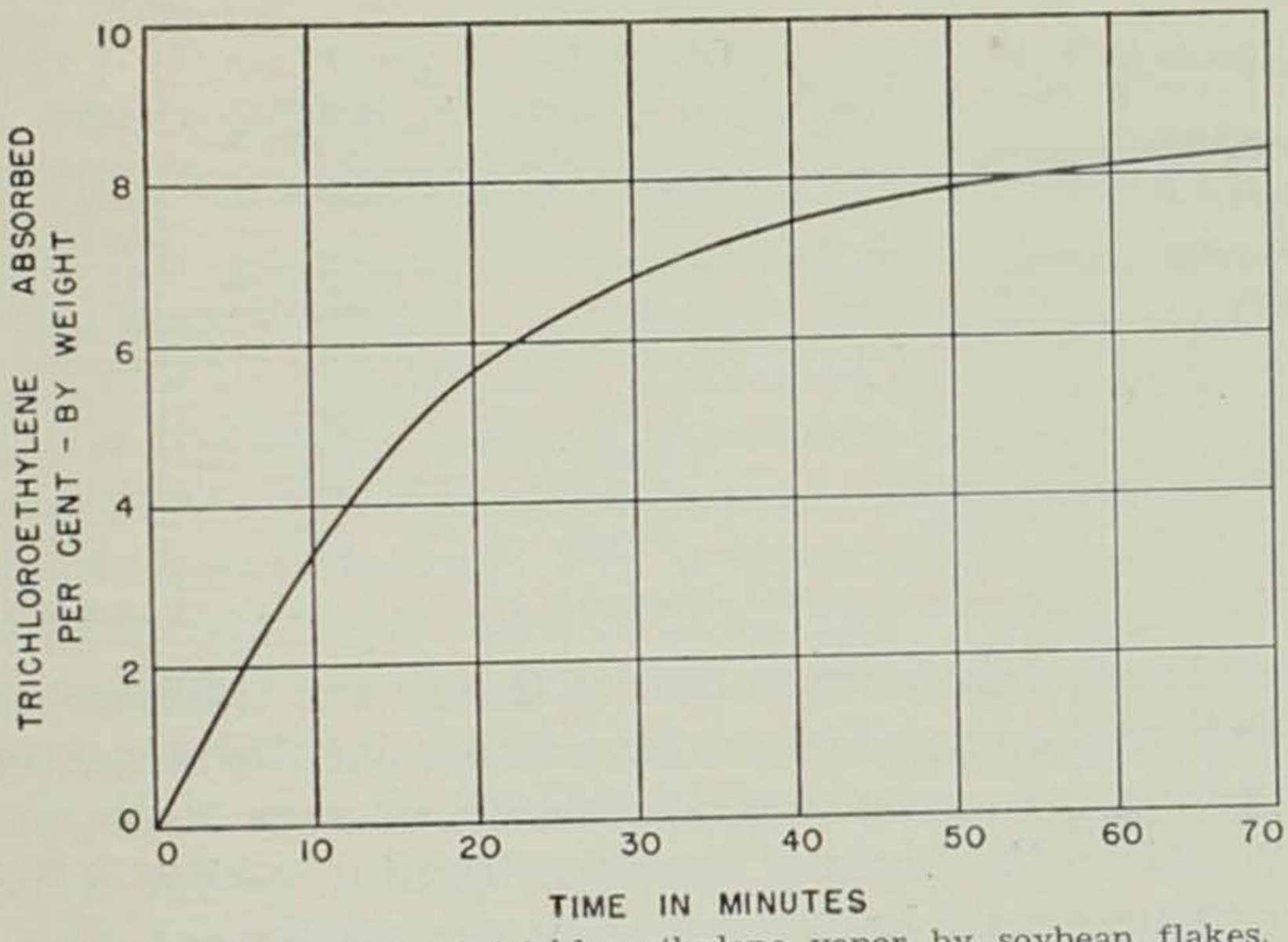

Fig. 18. Absorption of trichloroethylene vapor by soybean flakes.

and extracted soybean meal was then studied (37). Thirty gram portions of flakes were placed in evaporating dishes in tight cans with trichloroethylene in such a manner that the liquid solvent was not in contact with the flakes. They were allowed to stand for three days after which the flakes were weighed, the increase in weight of the flakes being taken as the amount of trichloroethylene absorbed. The absorption for extracted meal was 0.064 gram per gram of flakes; for unextracted flakes 0.495 gram per gram.

Fifty grams of flaked soybeans were placed on a screen ¾ inch above the bottom of a small sheet metal can. Air was bubbled through trichloroethylene at a known rate and run into the bottom of the can below the screen. The can and its contents were weighed at 10 minute intervals to obtain the amount of trichloroethylene absorbed. These results are shown in Fig. 18.

As a further check the trichloroethylene vapor and air mixtures were blown through a column of flakes 3-feet long by 3-inches in diameter for periods of 30, 60, and 90 minutes at rates of air flow from 0.03 to 0.07 cubic foot per minute. The resulting data are given in Table 17. As a result of these experiments the condensers were vented into the incoming flakes through a pipe extending from the upper side of the outlet end of the condenser to the side of the flake transition below the hopper. Air in the condenser saturated with trichloroethylene vapor passed upward

TABLE 17
ABSORPTION OF TRICHLOROETHYLENE BY SOYBEAN FLAKES

Rate of flow ft. per min.	Density of flakes lb. per cu. ft.	Trichloroethylene Absorbed		Time Min.
		lb. per lb. of flakes	percent of total in air	
2.6	18.6	0.04	92.8	90
3.0	15.3	0.04	79.9	60
3.0	15.0	0.01	54.1	30
1.8	20.3	0.01	46.2	60

into the downward moving mass of flakes, the trichloroethylene vapors being absorbed, and the air passing out. No measurements of the volume of air leaving the condenser were made, but it is evident that on the basis of the experimental work, a 3-foot column of flakes would have a capacity several times that needed. Since fresh flakes were constantly being added to the downward moving column, it was equal in effect to a column several times as great as the nominal length. In addition to the vapor vented from the condenser the column also acted as a moving seal to prevent solvent vapor from escaping from the extractor. Under normal operating conditions, no odor of trichloroethylene could be detected in the hopper above the flakes.

General Operation of the Unit

After making the changes discussed the pilot-plant unit operated very satisfactorily. Extraction rates as high as 160 pounds per hour were attained although this was above the capacity of the driers to remove all of the solvent. The normal rate of operation with good flakes was about 125 pounds per hour or 1.5 tons per 24 hours. The residual oil in the meal under good operation was less than 1 per cent. The solvent loss was about 10 pounds per ton of beans. The data obtained from the operation of the plant was considered adequate for the design of a commercial unit.

COMMERCIAL PLANT OPERATION

Introduction

Based on the data secured in the pilot plant studies, a plant was designed to have a processing capacity of 10 tons of beans per day. An agreement was entered into between Iowa State Col-

lege and J. Roach Sons, Inc., Plainfield, Iowa, in which the Iowa Engineering Experiment Station furnished the necessary design and specifications for the construction of the plant and the technical direction of its operation, until in satisfactory production. Since the plant was built under war-time restrictions the only new equipment which could be purchased were the extractor, the driers, and the stripper, which were built to specifications. This made necessary the utilization of some used equipment, part of which was unsatisfactory. The original stripper was later replaced by a combination of a climbing-film evaporator, filter, and packed column. The evaporator and filters were built by the plant mechanics from Station drawings and the packed column was constructed in the Engineering Experiment Station shop. As usual in process plants of new design, some difficulties in operation made certain changes and adjustments necessary.

Plant Operation and Equipment

A flow sheet of the plant is shown in Fig. 19 and the general arrangement of equipment in Fig. 20. The operation of the plant was as follows:

The beans were dumped from trucks into a concrete pit having a capacity of about 3 tons. The bottom was slanted to feed into a bucket elevator which was used to convey the beans to an overhead bin of about the same capacity as the pit. A magnetic separator to remove tramp iron was located in the spout between the top of the elevator and the upper bin.

The beans from the overhead bin were fed by gravity through a vibrating feeder to a single pair of cracking rolls. This was satisfactory for dry beans but not for beans containing 13 to 14 per cent moisture so that the single pair of rolls was subsequently replaced by a 2-pair high set of rolls with a LaPage cut. The beans were cracked into six or eight pieces and dropped into a bucket elevator which fed them to the conditioner. The conditioner was a steam-jacketed screw conveyor which heated the beans to about 65°C. (150°F.) before dropping the cracked beans onto the flaking rolls. The first rolls used for flaking were 10 inches in diameter but these were too small for satisfactory operation and were later replaced by a pair 20 inches in diameter by 24 inches long revolving at about 200 r.p.m. The cracked beans were rolled into flakes about 0.010 inch thick.

The flaked beans fell from the flaking rolls into a screw conveyor and then into a bucket elevator which dropped them into the extractor. Some breaking of the flakes occurred in the elevating process.

The combined extractor and drier consisted of a continuous Redler chain conveyor travelling in a steel casing connected in the shape of an inverted trapezoid with rounded corners. The parallel sides were set at a slight angle with the horizontal, the flake inlet end being the higher. The chain was driven at about one foot per minute by an electric motor operating through a speed reducer and a rachet drive. The lower casing which served

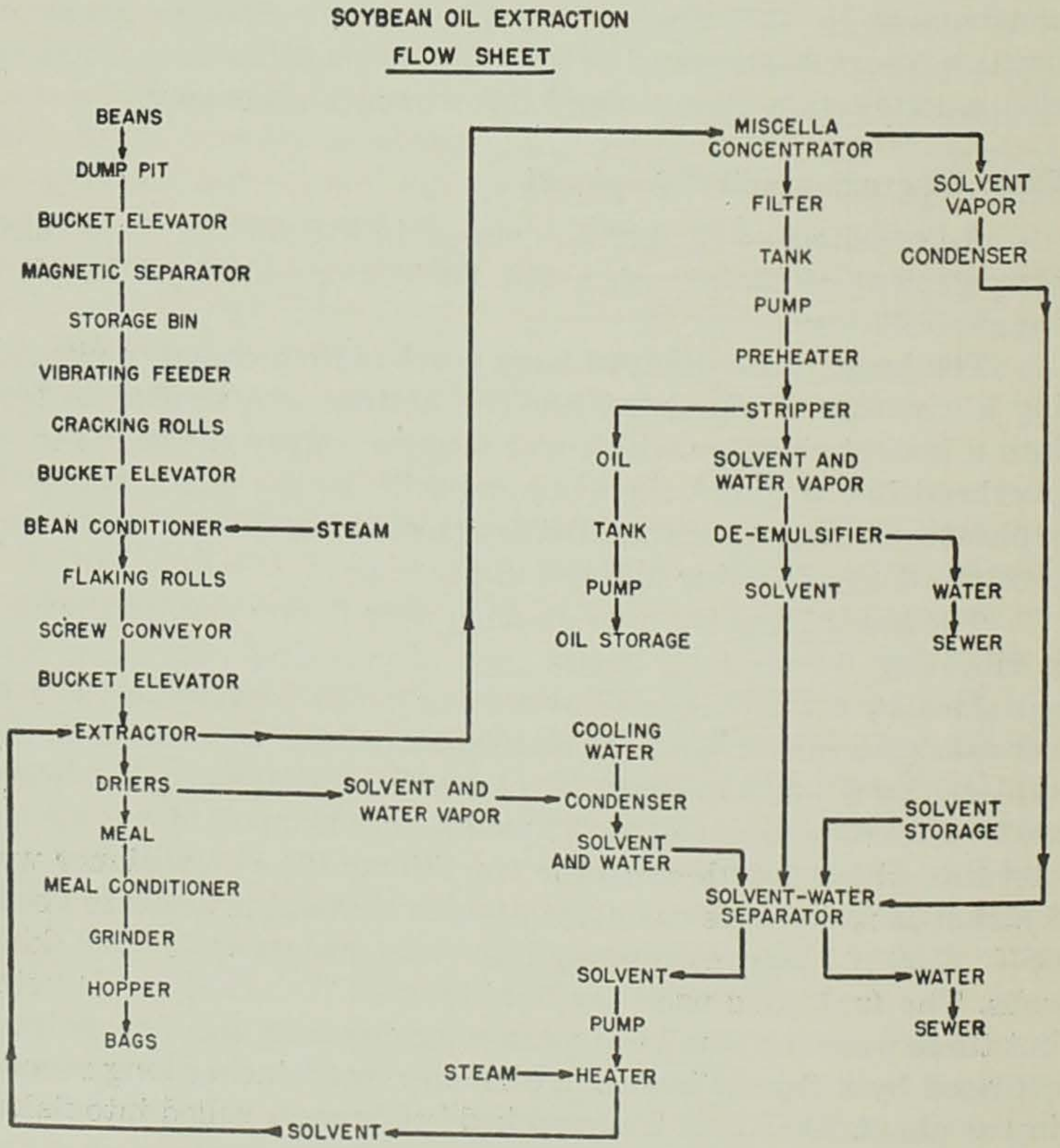

Fig. 19. Flow sheet of the soybean extraction apparatus (Plainfield).

as the extractor element had a cross section 8 inches by 12 inches and an effective extraction length of about 30 feet.

The rate of processing of the flaked beans could be varied by changing the speed of the Redler chain or by altering the type of flakes used. For a given type of flake the processing rate varied directly with the chain speed. Since thin flakes are more bulky than thick ones, less of the thin flakes by weight may be processed at a given chain speed than thick flakes. Other variations in the flakes due to bean characteristics such as age and moisture may affect capacity. Beans having up to 14 per cent moisture (the maximum which can be safely stored) were processed satisfactorily, the moisture content having no apparent effect on extraction efficiency. The maximum rate of chain travel depended largely upon the resistance of the flakes to solvent flow.

A column of flakes about 30 inches high was maintained over the extractor to prevent the escape of solvent vapor. This level was maintained by feeding a slight excess and allowing the excess to overflow into a bin. When the bin began to fill the feed rate to the cracker was reduced and the flakes from the bin were fed by gravity into the flake conveyor.

The solvent was pumped from the separator tank through the heater by a rotary pump and into the end of the extractor opposite the flake entrance. The heater consisted of a steam-jacketed pipe which raised the solvent's temperature to 65°C. (150°F.). The flow of solvent was regulated by a needle valve, the rate of flow being determined by a rotameter. The direction of flow was opposite to that of the flakes, thus producing counter-current extraction.

The solvent flow was adjusted to produce a miscella containing about 20 per cent oil. This required about 11 gallons of solvent per 100 pounds of beans of which about one-half became part of the miscella, the balance being carried out of the extractor and into the driers with the meal. When the thickness of the flakes was maintained at approximately 0.010 inch and the miscella at 20 per cent oil concentration the oil content of the dried meal averaged between 0.7 and 0.8 per cent.

Originally the miscella was concentrated and the solvent stripped from the oil in a falling-film stripper similar to that used in the pilot-plant installation. It was difficult to completely strip the oil in this unit, complete stripping involving overheating of the

oil, causing it to be dark colored. The miscella was not filtered prior to running to the stripper and the tubes become clogged with fines necessitating frequent cleaning. Because of these difficulties the stripper was replaced by a combination of a climbing-film evaporator, a filter, and a packed-column stripper.

In the new stripping arrangement the miscella, containing 20 per cent oil and 80 per cent solvent, flowed out of the extractor through screens in the side just below the point of entry of the flakes and into a rectangular tank in a pit below. The bottom of this tank was connected to 6 vertical steam-jacketed 1-inch pipes. These vertical pipes were connected at the top to a steel flash chamber by means of long sweep elbows. As the miscella entered the hot vertical pipes part of the solvent was vaporized, forcing the remainder of the miscella upward at high velocity into the flash chamber where additional solvent vaporization occurred. This resulted in concentrating the miscella from 20 per cent to 80 per cent oil content. The solvent vapor passed into a vapor line in the side of the flash chamber and went to a water-cooled surface condenser where it was condensed, the liquid solvent flowing by gravity into the solvent-water separator tank.

The concentrated miscella from the flash chamber flowed out of the bottom by gravity into a leaf-type enclosed filter and continued by gravity into a small tank in the pit below the extractor. Pressure filtering would doubtless have resulted in a longer filtering cycle and a more completely filled filter, but was not practiced in order to keep equipment and operations as simple as possible. About one pound of fines per ton of beans processed was recovered in the filters. Since the oil content of the fines was approximately 50 per cent of the total, the oil-free solids were equivalent to approximately one-half pound per ton. The length of the filter cycle varied, a change of filters being required every one to four days, with high moisture beans apparently producing a shorter cycle. Corn mixed with the beans also shortened the filter cycle, the starch from the corn plugging the filter cloth. Cleaning the filters was done by scraping off the cake and washing the leaves with solvent.

The filtered miscella ran by gravity into a small tank from which it was pumped through a preheater to the top of the packed-column stripper. Here the hot miscella trickled down over a bed of Berl saddles heated by vertical steam pipes. Steam was also

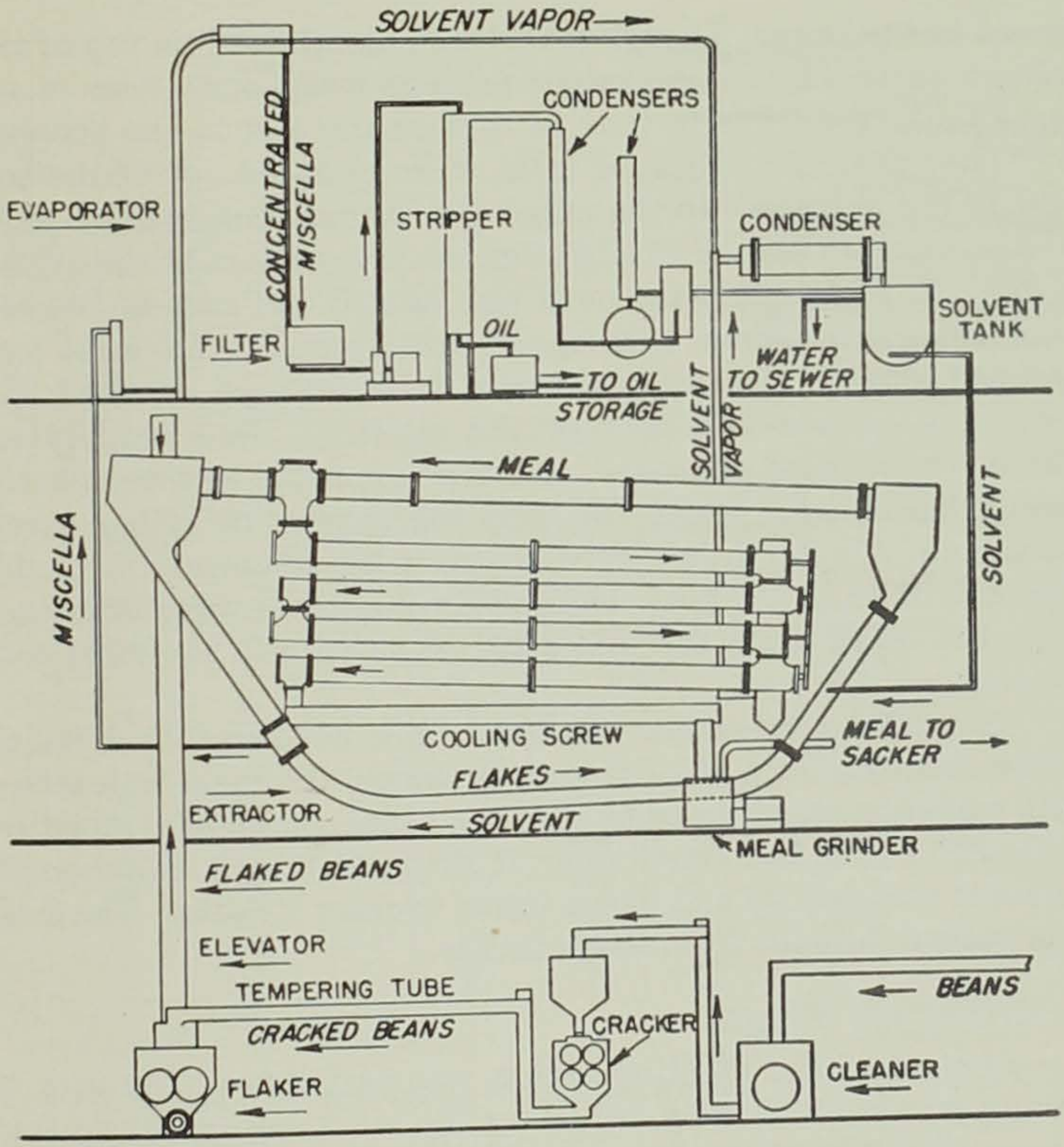

Fig. 20. Diagrammatic layout of the commercial apparatus of Fig. 21.

blown into the bottom and up through the saddles sweeping out the last traces of solvent. About one pound of steam per pound of oil were required. The oil flowed out the bottom through a trap into a small tank from which it was pumped periodically to an outside storage tank.

The solvent vapor and water vapor from the top of the stripper passed into a condenser where they were partially condensed. The mixture of liquid and vapor then went into an enclosed tank attached to the reflux condenser and finally to the solvent-water separator tank. This tank was a rectangular galvanized iron tank having a capacity of about 25 gallons. Here the solvent settled to the bottom where it was drawn off and the excess water over-

flowed to the sewer. Enough water was maintained on top of the solvent to prevent evaporation. Solvent was added from a reserve tank from time to time to replace any lost in the process.

The extracted flakes, or meal, were elevated out of the solvent in the extractor to the upper nearly horizontal steam-jacketed portion of the loop constituting the first section of the drying system. In this section the meal was heated and part of the solvent vaporized. From this upper drier section the meal was dropped into the first of two lower drier sections in which the remaining solvent and part of the moisture were evaporated. These lower driers consisted of 1-foot diameter steam-jacketed steel tubes through which the meal was carried by ribbon screw conveyors. All the driers were heated with steam at full boiler pressure of 90 to 100 p.s.i. The solvent-free meal was discharged from the lower drier through a barrel valve into the meal conditioner.

The meal conditioner consisted of a cut-and-folded 8-inch screw conveyor in a steel trough in which the meal was mixed with sufficient water to bring it to the usual commercial moisture content.* From the conditioner it dropped into a hammermill where it was ground and blown into a cyclone collector. The meal was sacked directly from the collector.

Control of the Process

Control of the plant operation was as follows: The rate of flaking was controlled by adjusting the vibrating feeder so that a very slight overflow was maintained. The flow of solvent was set by adjusting a needle valve in the line to give the proper reading on the flow meter to correspond with the rate of flake feed. This was checked hourly by determining the gravity of the miscella flowing out of the extractor by means of a hydrometer. The oil was tested for solvent by the pyridine-sodium hydroxide test. Thickness of the flakes was determined by micrometer calipers. In addition to the above routine control tests a large number of determinations were made on the oil content of the meal using the Sohxlet extraction method. Experience indicated that if the proper flake thickness and miscella gravity were maintained then

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* The moisture content of soybean meal is commonly adjusted in all processing plants to about 11 per cent. The authors believe that there would be certain advantages in selling the meal without the addition of water, on a dry basis. This would eliminate the possible development of mold resulting from damp spots due to imperfect mixing of the added water and would reduce freight costs.

TABLE 18
STEAM CONSUMPTION OF THE COMMERCIAL PLANT

Equipment	Pounds per hour (basis of 15 tons beans per 24 hours)	Pounds per ton of beans
Redler chain drier	125	200
Upper screw drier	176	282
Lower screw drier	155	244
Evaporator	175	280
Stripper—Indirect	55	88
—Direct	225	360
Bean conditioner	50	80
Total steam per ton		1554
Total steam per hour	961	
Total boiler horse power	961 ÷ 34.5 = 27.9	

TABLE 19
COMMERCIAL EXTRACTION PLANT OPERATION DATA*

Run No.	Time of operation hours	Beans processed per 24 hrs. in tons	Moisture content of beans—per cent	Solvent flow gal. per hr.	Oil in miscella —per cent	Oil in meal —per cent	Solvent loss lb. per ton of beans	Coal per day tons	Flake thickness inches
1	24	11.9	—	100	18.8	1.10	—	—	0.016
2	21	14.2	—	106	20.0	1.50	—	—	0.017
3	88	12.0	12.6	109	20.3	—	10.3	1.5	0.010
4	45	11.9	13.7	108	20.0	0.80		1.6	0.009
5	15	12.5	13.2	116	21.0	0.75	12.8	1.6	0.009
6	69	13.6	13.1	130	20.7	0.70		1.6	0.009
7	134	14.0	13.1	130	20.5	0.73	—	1.7	0.009
8	126	14.2	13.1	130	20.5	0.88	10.4	1.6	0.011
9	110	14.4	13.0	130	20.5	0.85	7.7	1.7	0.010
10	136	14.6	—	130	20.0	0.67	7.5	1.5	0.009
11	132	14.0	—	135	21.0	0.67	8.9	1.5	0.010

* Average data for time indicated.

the oil content of the meal would be known between reasonable limits.

Steam and Power Requirements

Steam for the plant was furnished by a stoker-fired coal-burning boiler of the horizontal fire-tube type with a maximum allowable steam operating pressure of 100 p.s.i. The steam consumption for the various units of the plant is given in Table 18. These data were taken when operating at 12 tons per day and recalculated for a rate of 15 tons per day. In addition to furnishing process steam the boiler also supplied space heating steam when needed. The building was heated by two unit heaters with forced air circulation and by heat losses from the equipment. The steam required for this is additional to the data given in Table 18 as the steam consumption data were secured in the summer. The coal consumption data (Table 19) indicate an evapora-

TABLE 20
APPROXIMATE ANALYSIS OF COAL

	As received	Dry basis
B.t.u.	12,250 per lb.	13,386 per lb.
Moisture	8.5%	
Ash	7.5%	8.2%

tion in the boiler of approximately 7 pounds of water per pound of coal. An analysis of the coal used is given in Table 20.

Electric power was purchased. Power consumption was about 43 kwh per ton of beans with an additional consumption of about 4 kwh per ton for lighting. Water was pumped by an electrically operated pump, the power used for this purpose being included in the above figures.

Labor Arrangements

Two men per 8-hour shift, in charge of a plant manager, handled the regular operation of the plant. Technically trained men were not used for this work. A truck and driver were utilized to haul beans from storage to the plant but with improved storage facilities this man could be eliminated. The beans were purchased, dried to a maximum of 14 per cent moisture (if above 14 per cent moisture), and stored at the main elevator of the company. The men on the regular operating shift assisted in the loading of trucks with meal and handled the pumping of oil into tank cars. They also did the routine cleaning of floors and equipment, ordinary repairs, and any special week-end cleaning of equipment.

Data on the various items entering into processing costs are given in Table 21. Actual dollar costs of such items as labor, coal, and power are not given as these will vary because of location or time and can be calculated for specific cases from the data given. Any process of oil removal involves some manufacturing loss because of the difference in the amount of moisture in the beans and the meal. It is customary in the trade to readjust the moisture content of the meal to approximately the equilibrium value. Moisture losses for meal of different moisture contents produced from beans containing 14 per cent moisture (the moisture basis on which beans are purchased) are shown in Table 22. The average moisture content of the meal produced was 12.8 per cent representing a loss due to moisture of 4.0 per cent.

Results of analyses of typical samples of the oil produced are summarized in Table 23. Both oil and meal were satisfactory, being accepted readily by the trade. The oil content of the meal averaged below one per cent, and it had a pleasing odor and taste.

TABLE 21

AVERAGE COST DATA—COMMERCIAL PLANT

Item	Per ton of beans
Labor[1]	3.51 man hours
Management[2]	0.75 man hours
Coal[3]	0.11 ton
Power (electric)[4]	44 kwh
Lights[4]	4 kwh
Solvent[5]	8.6 lb.
Depreciation[6]	$0.89
Taxes and insurance[7]	$0.22
Repairs and Supplies (est.)	$0.10

[1]No labor for hauling beans. 2 men per shift per day 48 hours ÷ 15 tons =3.3 man hours + 10 man hours per week. Starting and stopping or 0.12 hours + 8 man hours per week repairs, boiler cleaning, etc., or 0.09 hours.

[2]Manager 48 hours per week = 0.6 man hours + 0.15 man hours per ton to main office buying beans, selling meal, etc.

[3]Amount in Table 19 plus 10 per cent for line losses.

[4]From meter readings.

[5]Three months average.

[6]10 per cent of $30,000 for equipment, 5% of $20,000 for building and bean storage for 4472 tons.

[7]Calculated at 2.5 per cent.

TABLE 22

MANUFACTURING LOSSES DUE TO MOISTURE*

Moisture content of finished meal —per cent	Manufacturing Loss	
	lb. per ton	per cent
8	129	6.5
9	127	6.4
10	125	6.3
11	108	5.4
12	93	4.7
13	77	3.9
14	62	3.1

* Assuming a moisture content of 14 per cent in beans.

TABLE 23

CHARACTERISTICS OF TYPICAL SAMPLE OF OIL PRODUCED

Free Fatty acids	0.35 to 0.50%
Gardner break	0.40 to 0.51%
Moisture & volatile	0.17 to 0.26%
Solvent	Less than 0.02%
Refining loss	4.1 to 6.1%
Color—refined	Yellow 70; Red 11
—bleached	Yellow 18; Red 1.8

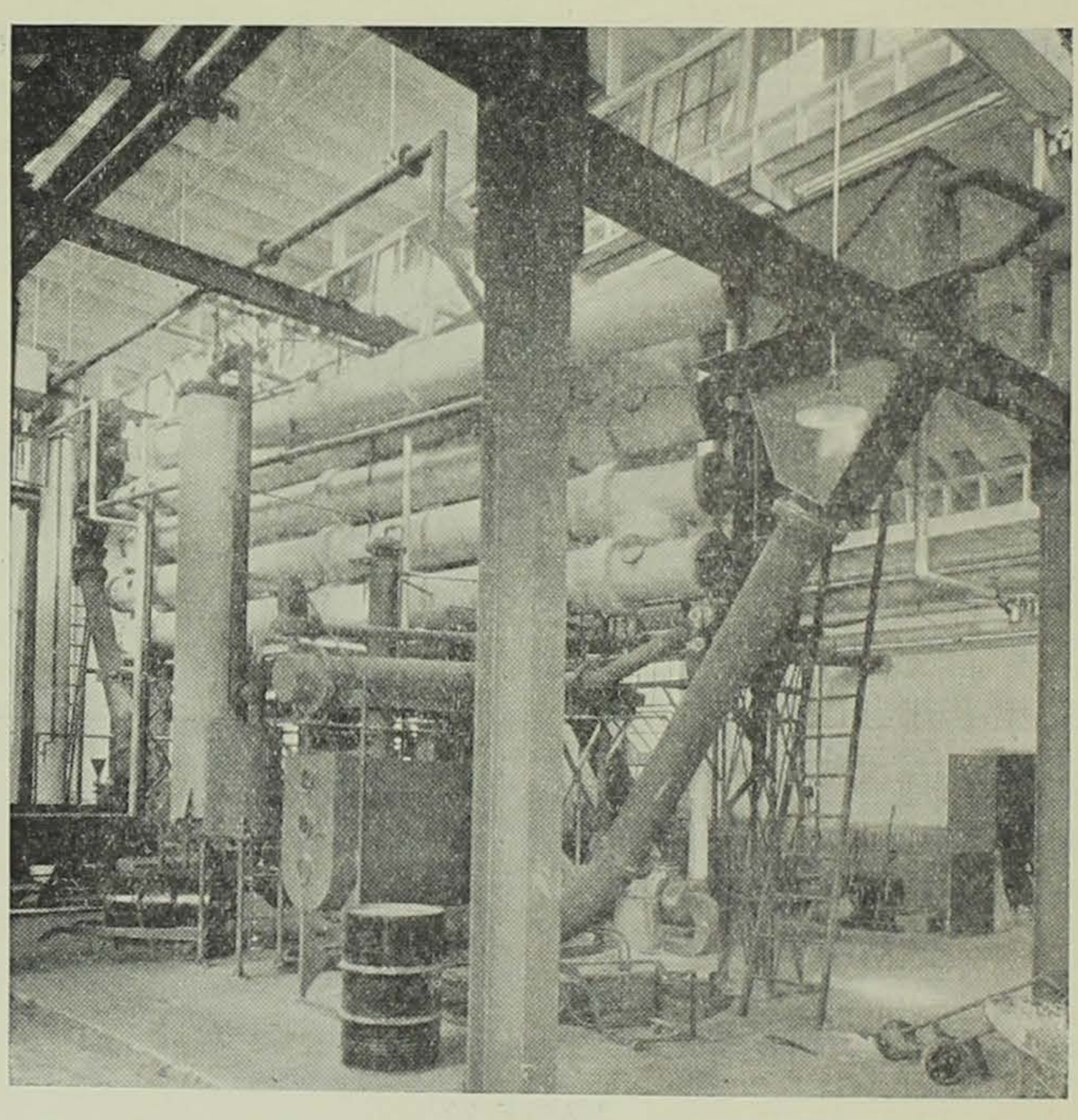

Fig. 21. Photograph of the commercial equipment developed by the Crown Iron Works.

FURTHER DEVELOPMENTS

Commercial equipment, embodying the principles developed in this work, is being manufactured by the Crown Iron Works Co., Minneapolis, Minn., under license from the Iowa State College Research Foundation. Fig. 21 shows the general layout employed in a plant designed to process 25 tons of soybeans per day.

SUMMARY

The commercial extraction plant designed to process 10 tons of soybeans in 24 hours satisfactorily processed 15 tons of beans containing up to 14 per cent moisture. The steam used was 1673 pounds per ton, the power 44 kilowatt-hours per ton, the labor (not including storage handling) 3.7 man-hours per ton, and solvent loss 10 pounds per ton. The meal contained less than one per cent oil and the oil satisfactorily met commercial standards.

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APPENDIX A

Analytical and Control Methods

The analytical methods used in these studies were standard methods with the exceptions and modifications as noted below.

Soybeans

Moisture: Five to ten grams of flaked beans were heated at 130°C. (266°F.) for 3 hours, (19), cooled in a desiccator, and weighed. The difference in weight was taken as the moisture content.

A somewhat faster means is an adaptation of the Dean and Stark method for determining water in lubricating oils (29) (8) (1) in which 10 grams of beans and 200 ml of xylene are placed in a 500 ml flask connected to a Dean-Stark trap and a reflux condenser. Boiling for 20 minutes vaporizes the water which together with the xylene collects in the trap, any excess xylene on top of the trap running back into the flask.

The amount of water is read from the graduated tube on the trap. This method should be very satisfactory for control purposes. Routine moisture determinations on the beans for the commercial unit were made on the whole beans in a Steinlite electrical moisture apparatus.

Oil: The Sohxlet extraction method was used. The usual procedure (19) calls for extracting ground beans for 2 hours after which the extracted beans are ground in a mortar and extracted for a second 2-hour period. Since

TABLE 24

OIL EXTRACTED IN ONE HOUR FROM FLAKED BEANS, AND IN FOUR HOURS FROM GROUND BEANS

Sample No.	Flaked Beans		Ground Beans		Difference in oil extracted, per cent
	Moisture per cent	Oil extracted, dry basis, per cent	Moisture, per cent	Oil extracted, dry basis, per cent	
1	9.73	21.7	8.66	22.4	0.7
2	12.3	21.3	9.17	22.1	0.8
3	11.0	21.9	10.5	22.8	0.9
4	8.88	22.4	9.73	23.4	1.0
5	9.73	22.9	9.76	23.8	0.9
6	12.2	21.0	11.8	21.8	0.8

excellent extraction results can be secured using flaked beans, checks were made to determine the feasibility of extracting flaked beans without regrinding. Samples of beans were analyzed for oil by the above method and samples of the same beans were flaked to an average thickness of 0.016 inch and then extracted for 1 hour. Commercial hexane (Skellysolve B) was used as a solvent in the usual Sohxlet extraction apparatus. The results (Table 24) on a series of 6 samples showed an average of 0.85 per cent less oil removed from the flaked beans. Flakes which had been extracted one hour were then extracted one additional hour with fresh solvent removing approximately 0.6 per cent more oil. Hence 2-hour extractions of flaked beans should give results within about 0.3 per cent of those by the conventional method. Flakes used in a solvent extraction plant should on the average be thinner than those used in this work, ordinarily 0.010 inch or less. Results using these thinner flakes will show less discrepancy in comparison with the standard method. For greater accuracy the extraction time was subsequently set at 3 hours rather than 2 hours.

The detailed procedure used was as follows: 5 gram samples of flaked beans were weighed and placed in an extraction thimble and covered with a wad of absorbent cotton. The thimble was placed in a medium sized (38 mm) Sohxlet extractor connected to a reflux condenser and a 150 ml flask containing 100 ml of hexane heated by an electric hot plate. The extraction was continued for 3 hours after which the flask was disconnected and connected to a condenser. The solvent-oil solution was concentrated by boiling to about 10 ml after which the flask was removed and placed on a steam hot plate and left there for 45 minutes after most of the solvent appeared to have evaporated. The flask was cooled in a desiccator and weighed.

Thickness of Flakes: Thickness of the soybean flakes was determined by means of micrometer calipers.

Oil: Standard methods for tests of oil quality were used (40).

Trichloroethylene in the Oil

Specific Gravity Method: The specific gravity of trichloroethylene at 25°C. (77°F.) is 1.46 that of soybean oil at 0.92. The relation between specific gravity and composition of mixtures of the two is given in Fig. 22. The most

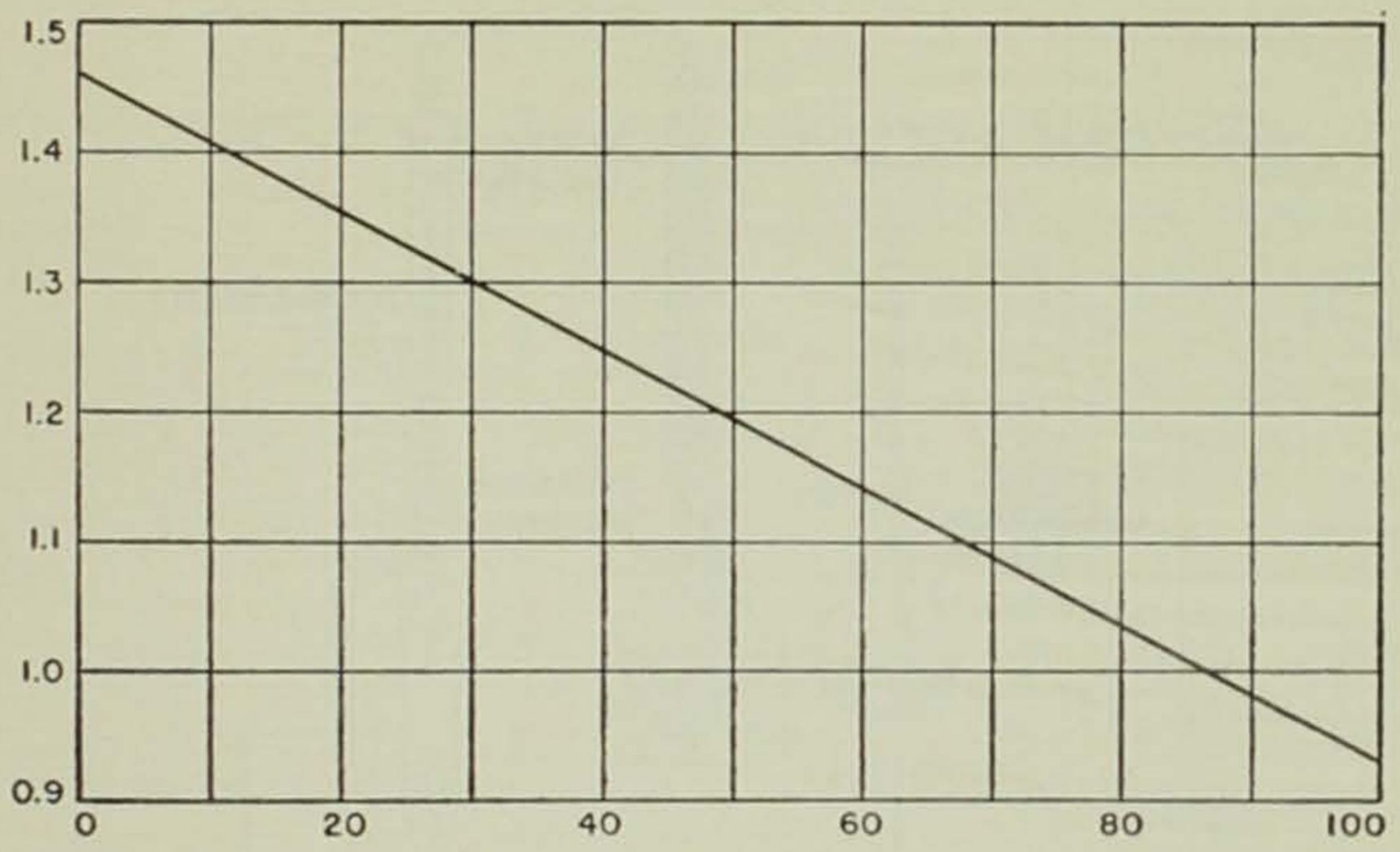

Fig. 22. Specific gravity of trichloroethylene-soybean oil mixtures.

practical method for determining the composition of miscella from either the laboratory pilot plant or the commercial plant was by means of a specific-gravity determination. For most of the laboratory work the gravity was determined by a Westphal balance and in the plant by a hydrometer. This method is satisfactory for amounts of trichloroethylene above 10 per cent. Below 10 per cent the solution becomes too viscous for accurate results unless the temperature is raised to about 50°C. (122°F.).

Steam Distillation Method: Since trichloroethylene may be removed from solutions in oil by steam distillation, Measamer (37) in this laboratory investigated the possibility of determining the trichloroethylene content of oil by a steam-distillation procedure. The apparatus used is shown in Fig. 23. The oil sample was heated in a bath which was maintained at 100°C. (212°F.) and steam bubbled through the sample. The steam and solvent vapor condensed and ran into the receiver where the trichloroethylene went to the bottom, the excess water overflowing at the top. When no more drops of trichloroethylene could be observed falling through the water, the distillation was stopped and the volume of the solvent observed. The total amount of water condensed with the solvent was also determined, from which a correction for water-dissolved trichloroethylene could be made and added to the observed amount. The solubility of trichloroethylene in water at 25°C. (77°F.) is 0.11 per cent.

To check the above procedure, known amounts of trichloroethylene were added to solvent-free soybean oil, and the mixtures steam-distilled. The results for a series of 8 samples each containing approximately 3 per cent solvent are given in Table 25. The accuracy for the method should increase for higher concentrations of trichloroethylene and decrease for lower concentrations.

This method is more time consuming than the specific gravity method and probably no more accurate for amounts of trichloroethylene above 10 per cent.

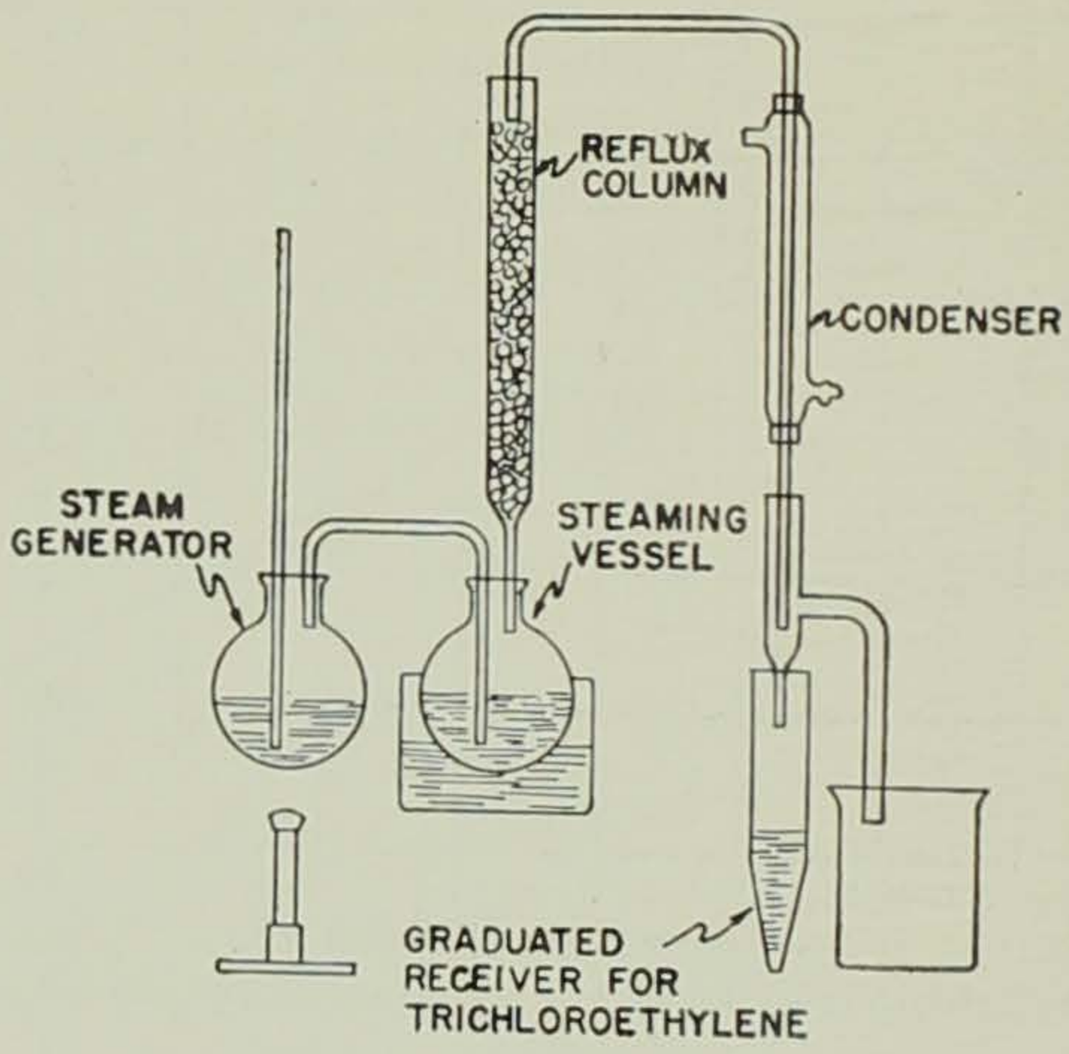

Fig. 23. Steam distillation apparatus as used by Measamer (37).

Spectrographic Method: Work in this laboratory by Yates (55) has shown that amounts of trichloroethylene between 0.1 and 1.0 per cent can be determined by means of a spectrograph with an accuracy of 7 per cent. The sample of oil was placed on a copper wire which was held in a flame thus producing copper chloride which gave distinct bands between the wave lengths of 4259 and 4518 Angstrom units. The concentration of trichloroethylene in the oil was determined from the length of time the bands were distinct in the spectrum, usually between 2 and 8 seconds. It was necessary because of differences in color perception, for each operator to draw his own curve of the time-per cent trichloroethylene relation.

Pyridine-Sodium Hydroxide Method: Fujiwara (14) described a method for detecting small amounts of chloroform. He heated to boiling a pyridine and 10 per cent sodium hydroxide mixture, the two liquids remaining as separate layers after heating. If a solution containing chloroform was added to the mixture the pyridine layer turned a blue-red. This laboratory adapted this test to trichloroethylene in soybean oil by adding 1 ml. of pyridine to 1½ ml. of 10 per cent sodium hydroxide in a test tube and heating, with shaking, to boiling. When a few drops of oil containing trichloroethylene were added, the pyridine layer turned a deep cherry red. The intensity of the color varied with the amount of trichloroethylene present thus making it possible for the reaction to be made into a quantitative method instead of a purely qualitative test. Work by Gilchrist and the authors resulted in the development of the method now used for routine quantitative determinations of small amounts of trichloroethylene in soybean oil. This method which is adaptable to other chlorinated hydrocarbons has also been used on methylene chloride-soybean oil mixtures.

The method is as follows: Add 1 ml. of pyridine to 1½ ml. sodium hydroxide solution (10 grams to 100 ml. of water) and heat to boiling in a

TABLE 25

DETERMINATION OF TRICHLOROETHYLENE IN SOYBEAN OIL BY STEAM DISTILLATION

No.	Per cent Trichloroethylene			Error per cent
	Added	Recovered	Corrected amount*	
1	3.25	2.594	3.016	7
2	3.25	2.594	3.016	7
3	3.25	2.432	2.854	12
4	3.25	2.432	2.854	12
5	3.25	2.594	3.016	7
6	3.25	2.821	3.25	0
7	3.25	2.756	3.18	2
8	3.25	2.594	3.016	7

* Amount recovered plus correction due to amount dissolved in water.

test tube. Add 2 drops of the soybean oil being tested. Allow the mixture to stand one minute for the color to develop and compare the depth of color with standard solutions.

The original color standards were made by adding oil containing known amounts of trichloroethylene to pyridine-sodium hydroxide solutions prepared as in the test. Since the color is not permanent, standards matching these were made up by mixing together in a water solution sodium dichromate and cobaltous chloride together with enough hydrochloric acid to give an acid reaction. The solutions were kept in test tubes sealed with paraffined cork stoppers. Standards were made for 0.02, 0.05, 0.1, 0.2, 0.3, 0.5, 1.0, and 2.0 per cent trichloroethylene. For amounts above 2 per cent trichloroethylene the oil should be diluted with solvent-free oil by a known percentage before applying the test.

The above method is the simplest and most accurate method found to date for determining traces of the solvent in the oil. Several precautions in the use of this method should be noted. Pyridine vapors are toxic and breathing them should be avoided so that the test should be carried out in a hood with a good draft. Pyridine is easily contaminated so as to give the red color. Test tubes of the reagent mixture allowed to stand in a room containing only traces of solvent vapor may show the test color upon heating. Chemically pure or reagent pyridine is satisfactory for the test. Some lots of so-called "practical" pyridine have been satisfactory, others sufficiently contaminated with a chloride to be useless. The dropper used for the oil should be rinsed out thoroughly with hexane or similar chlorine-free solvent after using.

Meal

Moisture: The method used for beans, that is heating to 130°C. (266°F.) for 3 hours, was used for determination of the moisture in the meal. In control work at the commercial plant a Steinlite electrical moisture apparatus was used. A special calibrated chart provided by the factory was suitable for the ground meal. A different calibration was necessary for the unground meal due to differences in apparent specific gravity.

Oil: The meal as it came from the driers or after it was ground to commercial meal size was extracted in a Sohxlet apparatus with hexane using the same procedure as for beans except that a 1-hour extraction time was used.

Trichloroethylene in Meal: The following method which is an adaptation of the pyridine-sodium hydroxide method used for oil was used for traces of trichloroethylene in the meal. Allow the meal to cool in a tightly stoppered bottle and then weigh out approximately 10 grams rapidly. Add the weighed meal to a 125 ml. flask containing 40 ml. of hexane, stopper tightly and allow to stand for a few minutes. In a test tube add 1 ml. pyridine to 1½ ml. of 10 per cent sodium hydroxide solution and heat to boiling. Add 4 drops of the hexane from the flask containing the meal. Compare the depth of color after 1 minute with the color standards (standards prepared similarly to those for oil, but calibrated for the meal test).

A simple qualitative test accurate for small traces of the solvent in the meal is to taste it. The meal should be chewed for about a minute. The solvent gives a characteristic sweetish taste accompanied by a cooling sensation.

The determination of large amounts of trichloroethylene in the presence of moisture in the meal may be done as follows: Weigh two samples of the meal in covered weighing pans. Remove the cover from one and heat at 130°C. (266°F.) for 2 hours in a drying oven then cool and weigh. Transfer the second weighed sample to a Dean-Stark moisture apparatus and determine the moisture. The difference in weight of the first sample minus the moisture removed from the second is the amount of solvent.

APPENDIX B

Results of Extraction of Oil from Frost-Damaged Beans

In September 1942, an early freeze occurred in a large part of Iowa, stopping the growth of many soybean plants and resulting in a considerable percentage of immature beans. Since many of the beans were designated as frost-damaged and subjected to heavy discounts in price, an investigation was made to determine the oil content of these damaged beans.* Results of oil determinations on 83 samples of beans from various parts of the state of Iowa and varying in damage from 0 to 88.8 per cent are given in Table 26. No consistent relation between damage and oil content is shown by these data, other factors such as variety and soil apparently obscuring any relationship which may exist. The variations in oil content are no greater than might be expected from a like number of samples of normal beans. It is interesting to note that a sample having 86.2 per cent frost damaged beans showed an oil content of 23.8 per cent leading to the thought that damaged beans might actually be higher in oil content than undamaged ones. To

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* Samples of beans were collected by R. C. Bentley, extension associate professor of Economics and Sociology, and various county extension directors. Grading of the beans was by H. A. Harlow, Grain Supervisor, Food Distribution Administration, U.S.D.A. (Cedar Rapids, Iowa). Cooperation by C. R. Weber, U.S.D.A. Agent in Agronomy at Iowa State College, and A. V. Tisher, Federal Licensed Grain Inspector, Des Moines, Iowa was also very helpful.

TABLE 26

OIL CONTENT OF IOWA'S FROST DAMAGED SOYBEAN CROP

County	Variety	Date planted	Oil content dry basis, per cent	Frost damage, per cent	Quality of beans as reported by County Directors
Floyd	McClave	5/25	19.5	88.8	
	Manchu	5/20	21.1	33.5	Medium beans
	Mukden	5/27	21.1	12.4	Good beans
Winnebago		5/17	23.8	86.2	Poorest in County
		5/17	21.7	7.1	Very good
Calhoun			19.9	59.3	
Poweshiek			21.1	59.0	
Howard			20.0	58.6	
Winneshiek			21.8	53.0	Poorest ½ County
		5/29	19.0	8.8	Best 20%
Black Hawk	McClave	5/23	19.4	52.7	
	Illini	5/26	20.9	27.0	
	Illini	5/16	21.0	15.4	
Chickasaw		5/15	21.5	52.0	
Clarke	Illini	6/25	23.1	51.7	
	Illini	5/28	22.6	18.2	
	Illini	5/20	21.4	16.2	50 bu. yield
Cerro Gordo	Mukden		19.9	53.1	Top Quality
Mitchell		5/30	20.2	48.0	Middle 30%
		6/15	20.7	7.3	Low 30%
		5/10	19.8	4.1	Top 30%
Linn	Illini	7/1	19.0	46.6	
	Illini	6/1	19.5	16.7	
Guthrie	Illini	6/6	21.1	43.0	
	Manchu	5/18	20.6	17.4	
	Mukden	5/25	22.2	0.6	
Hancock			21.3	40.0	40% Damage
		5/8	22.1	8.0	Good Quality
Taylor			21.0	36.7	Medium quality
	Dunfield	5/8	20.4	14.3	
Audubon	Mukden	6/15	20.2	34.4	
Kossuth	Mukden	5/25	20.4	30.0	Low 10-15%
	Richland	5/20	20.2	7.5	Top grade
	Mukden	5/20	21.5	5.0	Average quality
Grundy	Chief		21.4	29.5	
			19.9	9.8	
Pocahontas	Mukden		20.7	29.2	
	Mukden	5/12	21.5	18.5	Medium beans 12% M.
		5/10	21.3	1.4	Good beans 13% M.
Clinton			21.2	29.2	Late bean
			20.7	21.0	Average
			22.5	9.4	Early
Emmett			19.8	28.6	
			19.4	4.4	Top quality
Warren			23.6	28.6	
Bremer	Manchu	5/30	21.3	28.3	
		6/15	20.7	15.2	
	Mukden	5/15	21.3	9.4	

TABLE 26 (Continued)
OIL CONTENT OF IOWA'S FROST DAMAGED SOYBEAN CROP

County	Variety	Date planted	Oil content dry basis, per cent	Frost damage, per cent	Quality of beans as reported by County Directors
Carroll		5/20	22.9	26.4	
		5/20	20.3	14.4	
	Richland	5/15	19.0	0.0	
Buena Vista	Manchu	6/20	19.3	24.7	Low 10%
	Mukden	5/16	20.7	8.6	Best 10%
Dickensen	Mukden	5/15	20.9	24.1	
Polk	Mukden		20.3	23.5	
Tama		6/25	23.1	23.4	
		5/1	20.5	8.7	
		6/1	20.4	4.8	
Jones		5/26	20.4	22.0	
		5/29	19.2	5.2	
		6/6	21.2	2.3	
Woodbury		6/1	22.1	21.0	Poor
	Mukden		21.4	4.7	Fair quality
Johnson			21.5	20.5	Medium late
	Illini	5/25	21.8	9.9	
Wayne	Illini		20.6	19.2	
Sac			21.7	18.5	
			19.6	8.8	
Muscatine			21.2	17.0	
			20.4	2.0	
Humboldt		6/1	22.1	14.4	
		5/26	22.1	11.8	
Des Moines	McClave		18.4	10.8	
	Illini	5/22	22.2	4.2	
Ida		6/20	20.9	10.0	Poorest 30%
		6/2	21.2	1.7	Middle 30%
Cedar	Illini		20.0	7.8	
			19.4	3.8	11.7% Moisture
Hardin		5/27	19.7	4.6	
		5/25	19.0	3.5	
Butler			22.7	1.7	
Henry			21.6	1.0	
			21.7	.8	

* These samples were sent to the Federally licensed inspectors of central and eastern Iowa for grade determination. Furnished by H. A. Harlow's office in Cedar Rapids, compiled by Agricultural Economics Department, Iowa State College.

check this the oil contents of damaged, medium, and best beans from several lots of damaged beans were determined. The results (Table 27) show no consistent relation.

The oil from the damaged beans was decidedly green, and it was reported that this was very difficult to refine to produce the usual white hydrogenated-oil product. Processors also reported the oil harder to remove from the damaged than from the undamaged beans. To determine how well

the oil could be removed from the damaged beans, both damaged (23.5 per cent damaged) and undamaged beans were processed in an Anderson Midget Expeller. Using the maximum heat obtainable with the tempering apparatus the meal from the damaged beans contained 11.5 per cent oil and from undamaged beans 4.95 per cent oil. By preheating the beans so as to process at 124°C. (255°F.), it was possible to remove all but 4.86 per cent oil from the damaged beans. By increasing the temperature to 140°C. (284°F.), 25 per cent less power was used, and the meal had an oil content of 5.95 per cent. The oil had a dirty color with a greenish cast, the higher temperature producing poorer-appearing oil. The iodine number of the oil from the mature beans was 125.1 and from damaged beans 129.7 per cent.

TABLE 27

OIL CONTENTS OF DAMAGED, MEDIUM, AND BEST BEANS FROM CERTAIN SAMPLES

Variety	Oil, per cent (dry basis)			Damage %
	Damaged	Medium	Best	
Boone - 1	21.9	21.7	20.9	28.1
Boone - 2	21.6	22.0	20.7	30.7
Boone - 3	21.1*	21.4*	21.6*	56.8
Dunfield - 5	21.7*	21.9*	21.6*	20.8
Mukden - 5	21.0*	19.5*	20.8*	24.8
Richland - 5	22.5	20.3	19.3	4.3

* Extracted for 6 hours without regrinding. All other samples in above table were extracted for 2 hours, reground, and extracted for 1 additional hour. Because only small samples were available, the beans were ground with a mortar and pestle until they passed a 16 mesh screen.

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